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Abstract

The purpose of this work was to assess the effects of the amount of retained austenite content on the ductile-to-brittle transition temperature of martensitic precipitation strengthened stainless steels for four different precipitation strengthening systems, one utilizing NiTi strengthening and three utilizing R-phase strengthening. The retained austenite contents in the four systems were varied by varying composition. The austenite content in the NiTi strengthened system was varied by varying the chromium content and the austenite content in the R-phase strengthened systems was varied by varying the nickel content. The room temperature toughness levels of the NiTi strengthened system were quite low and it was decided not to pursue this system further. The three R-phase strengthened systems had sufficient room temperature toughness and strength to be of further interest. Of these three systems the primary focus was on the 12Cr/12Co/5Mo system. In this system four alloys, identical except for variations in nickel content, were the primary focus of the work. These alloys achieved, on tempering at 525°C for 3.16 hours, yield strengths on the order of 210ksi and ultimate tensile strengths of 235ksi. The effect of test temperature on the Charpy impact energy was investigated for two tempering temperatures for these four alloys. It was found for both tempering conditions that lower ductile-to-brittle transition temperatures were favored by increasing amounts of austenite in the structure. In fact, the ductile-to-brittle transition temperature was quite low, about -75°C, for the tempered at 525°C for 3.16 hours microstructure of the alloy in this series which contained the highest nickel and the highest amount of retained austenite after quenching. At this point it is believed the austenite content is an important contributor to the low ductile-to-brittle transition temperature of this microstructure. However, further work is required to determine the relative importance of retained and reverted austenite in controlling the ductile-to-brittle transition temperature in these types of steels.

The Effects of Retained Austenite on the Tensile Properties and Toughness of Ultra-High Strength Martensitic Precipitation Hardened Stainless Steels

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I. Motivation:

The ultimate objective of our work on ultra-high strength martensitic precipitation strengthened steels is the development of an ultra-high strength stainless steel for use in landing gear components. Such a material would have two attractive features. First, if the general corrosion resistance was sufficiently high then the components would not need to be cadmium plated as is presently the case with the low-alloy and high alloy steels used in landing gear. Second, if the steel has sufficient corrosion resistance then it is likely that the steel will be highly resistant to stress corrosion cracking. Designers and producers of landing gear such as Messier-Dowty have indicated that such a material would find immediate application.

II. Desired Properties of Such a New Steel:

First, such a steel should have an ultimate tensile strength of about 275ksi. While landing gear designers often claim to design to the ultimate tensile strength this is not the whole story. An acceptable yield strength is also required. A yield strength of 190ksi at an ultimate tensile strength of 275ksi would not be acceptable. Therefore, one should attempt to achieve yield strengths in excess of about 235ksi and yield strengths in excess of about 275ksi. In addition to strength the important properties to consider are general corrosion resistance, fracture toughness, ductile-to-brittle transition temperature, stress corrosion cracking resistance and fatigue. Achieving high fracture toughness does not appear to be a primary objective of landing gear manufacturers except for the case of Naval aircraft where a fracture toughness of 100ksi√in is presently required by the Navy. It would be desirable for the ductile-to-brittle transition temperature of the new steel to be below about -40°C. At the present time most landing gear are made of 300M and the fracture toughness of this ultra-high strength low alloy steel is about 75ksi√in. Therefore, it would appear reasonable to have as an objective a fracture toughness of 75ksi√in or higher. The K_{ISSC} of 300M is about 13ksi√in and that of the high alloy steel currently used in the landing gear of Naval aircraft is about 30ksi√in. Our objective would be to achieve a K_{ISSC} of at least 50ksi√in.

III. Background and History:

There is a long history of alloy development in this area and these activities have led to a number of steels although most of these steels are no longer produced or widely used. See Table I for a list of compositions. All of these steels are martensitic steels and one might separate them into four classes. The first class of such materials would be medium carbon secondary hardening steels containing about 12 to 16 wt.% chromium. The second class would be low carbon precipitation hardened steels containing no cobalt, such as PH13-8. The third class would be low carbon precipitation hardened steels containing cobalt. The fourth class would be steels in which strength is achieved by combining secondary hardening with other types of particle strengthening.

During the 60's and early 70's the development of martensitic ultra-high strength steels was an active area of research and was pursued by many specialty steel companies here, in Europe and in Japan. One approach was to use low carbon alloys and to achieve strength only by non-carbide

particle precipitation. There were a number of such grades at one time but all depended on precipitation of NiAl[1,2] and/or copper[1] to achieve high strength. Today the only such grades in use are PH15-5 which is strengthened by copper precipitation, Custom 450 which is strengthened by copper and Laves phase precipitation and PH13-8 which is strengthened by NiAl precipitation. PH15-5 and Custom 450 achieve maximum yield strengths of about 180ksi while PH13-8 achieves a maximum yield strength of about 210ksi. Later precipitation strengthened martensitic stainless steels began to utilize precipitation of NiTi [1,3] or Ni(Al,Ti) with or without copper additions. Examples of such steels are Custom 455 which can achieve a maximum ultimate tensile strength of about 245ksi but has a Charpy impact energy of only 9ft-lbs at this strength level and Inco 734.

Other low carbon martensitic high strength steels were developed to take advantage of age hardening observed in iron base alloys containing high levels of chromium, molybdenum and cobalt. Such alloys were Pyromet X-15 and Pyromet X-23 developed by Carpenter Technology; these alloys can achieve yield strengths as high as 237ksi. Concurrent with these efforts was the development of alloys which combined the age-hardening of martensite due to the combined additions of Cr, Mo and Co and the precipitation of NiTi. These alloys were developed in Germany and were designated Ultrafort 401, 402 and 403[4].

In addition, there have been attempts to combine secondary hardening with strengthening by other particle types. This work was pioneered in England[1] and involved the combined precipitation of NiAl and alloy carbides(secondary hardening). Finally, there was the work done by Crucible[4] which led to the development of the alloys AFC77 and AFC260. These are extremely complicated alloys which take advantage of secondary hardening and the age hardening obtained when large amounts of Cr, Mo and Co are simultaneously present. AFC77 has attractive levels of ultimate tensile strength but AFC77 has a very low yield strength in relation to its ultimate tensile strength and a low fracture toughness at the highest ultimate tensile strength. AFC260 has an ultimate tensile strength of about 260ksi and a maximum yield strength of about 220ksi and, of the alloys which have been investigated would seem to be the closest to satisfying the property requirements outlined above.

Today the only ultra-high strength martensitic stainless steel used in critical applications at yield strengths greater than about 190ksi is the steel PH13-8.

IV. Why Not Use Presently Available Steels:

For the proposed application one might consider using Custom 455, Pyromet X-15, Pyromet X-23, Ultrafort 401, Ultrafort 403, AFC77 and AFC260.

Custom 455 has a maximum yield strength of only 245ksi and while it has been reported to achieve fracture toughnesses as high as 79ksi√in, it has a room temperature Charpy impact energy of only 9ft-lbs which suggests that the ductile-to-brittle transition temperature determined from Charpy impact tests would be close to or above room temperature.

The steel Pyromet X-15 has a yield strength on the order of 215ksi, an ultimate tensile strength of 235ksi and a room temperature fracture toughness of about 35ksi√in.

The steel Pyromet X-23 has a yield strength on the order of 237ksi, an ultimate tensile strength of 258ksi and a room temperature fracture toughness of about 70ksi√in.

The steel Ultrafort 401 has a yield strength on the order of 227ksi, an ultimate tensile strength of 242ksi and a room temperature fracture toughness of about 95ksi√in. In addition, this steel has a ductile-to-brittle transition temperature as measured by DVM specimens which are similar to Charpy impact specimens but with a blunter notch, of less than -40°C.

The steel Ultrafort 403 has a yield strength on the order of 242ksi, an ultimate tensile strength of 245ksi and a room temperature fracture toughness of about 60ksi√in. In addition, this steel has a ductile-to-brittle transition temperature as measured by DVM specimens which are similar to Charpy impact specimens but with a blunter notch, greater than room temperature.

The steel AFC77 can have ultimate tensile strengths above 300ksi, but at this ultimate tensile strength it has low yield strength and low fracture toughness (see Figure 1)

The steel AFC260 seems to come the closest to satisfying the strength and toughness requirements. This steel can achieve yield strengths of about 220ksi at ultimate tensile strengths of about 260ksi and has fracture toughnesses in this strength range of about 60ksi√in to almost 80ksi√in, depending on the tempering temperature. The steel also can have $K_{I\text{SSC}}$ values of about 45ksi√in at this strength level. The question would be whether any additional strength could be obtained from this system by minor alloying additions. I do not see any way of doing this without reducing the martensite start temperature too much. An alternative is to utilize longer tempering times to enhance the strengthening by R-phase. One might increase hardness by 1 to 2 Rockwell-C by doing this.

V. Conclusions Based on Previous Work:

A. Phase Stability:

First, to achieve the desired yield strengths the alloys cannot contain large amounts of retained austenite. Therefore, the martensite start and finish temperatures cannot be too low. Second, it is generally felt that delta-ferrite in the structure can reduce strength and can have an adverse effect on the ductile-to-brittle transition temperature. Third, the smaller the austenite grain size, the smaller the martensite packet size, and the lower the ductile-to-brittle transition temperature. Therefore, preferred compositions would permit an essentially austenitic structure at fairly low temperatures while maintaining reasonably high martensite start and finish temperatures. These two requirements considered together place severe constraints on the compositions one might select given the necessarily high chromium contents.

B. Strengthening Mechanisms:

There are a number of strengthening mechanisms available. These include strengthening by particles of alloy carbides (secondary hardening), particles of the intermetallics NiAl and NiTi, particles of copper and strengthening by precipitation of particles when the alloy contains sufficient amounts of chromium, cobalt and molybdenum. All of these strengthening methods have been utilized in martensitic stainless steels.

While CoAl and CoTi are known to exist and to have the same crystal structure as NiAl and NiTi, their precipitation has not been observed in steels at aluminum and titanium levels on the order of one wt.%. However, one can achieve substantial strengthening in iron base alloys containing large amounts of cobalt (10 to 20 wt.%) and large amounts of aluminum and titanium (certainly more than about 3 wt.%) [5]. It is not clear such an approach to strengthening can be used in martensitic stainless steels because of the strong ferrite stabilizing tendencies of aluminum and titanium.

Strengthening by NiTi and NiAl has been studied extensively. Both compounds have the CsCl B2 structure and have lattice constants close to that of the martensite matrix. The misfit is greater for the NiTi than for the NiAl [1,6] and for this reason strengthening by NiTi is more effective than strengthening by NiAl when Ti and Al are present in the alloy at the same atomic percent. by varying the amounts of aluminum and titanium one can vary the composition of the Ni(Ti,Al) and the degree of misfit with the matrix. It is the authors experience that when NiTi and NiAl

strengthening are compared at the same strength level then NiTi has a more adverse effect on the ductile-to-brittle transition temperature than does NiAl.

A strong age hardening is observed in steels containing sufficient amounts of chromium, cobalt and molybdenum[7]. While the precipitates forming at low aging temperatures and short aging times associated with the strong age hardening response have not typically been identified, one study claims that these particles are of the R phase. The R phase identified in other work after aging at fairly high temperatures or long times at lower temperatures contains by weight about 25% chromium, 8% cobalt, 32% molybdenum and 35% iron[8]. While the particles associated with the strengthening do not appear to have been unambiguously identified we will refer to this strengthening mechanism as R phase strengthening. It is known that the degree of R-phase strengthening increases with both the cobalt and molybdenum contents in steels containing about 12% chromium. In fact the peak hardnesses which can be achieved after aging a low carbon steel containing 12 wt.% cobalt, 12 wt.% chromium, 4.5 wt.% nickel and 0.2 wt.% titanium for 4 hours are about Rockwell-C 42 at a molybdenum content of 2 wt.% and a Rockwell-C 50.5 at a molybdenum content of 5 wt.%[9]. The maximum hardness which can be achieved at the molybdenum level of 5 wt.% is about Rockwell-C of 55 after aging at 490°C for 83 hours[10].

Strengthening mechanisms are not always additive. For example, strengthening by copper precipitation and NiAl precipitation leads to almost the same yield strength as strengthening by NiAl alone, at least in one system we have studied. This effect appears to be partly due to the effect of copper on the kinetics of the NiAl precipitation. However, in general there is no reason to expect strengthening mechanisms to be additive. For example, if two particle distributions both increase the yield strength by the same amount when present alone it may be that when both are present the increase in the yield strength is essentially that due to one of the particle distributions or it may be that both particle distributions contribute to the strength so that the strengthening resulting from introducing both particle distributions is considerably more than the strengthening which could be achieved by either particle distribution alone. It is impossible to tell from the data available the extent to which strengthening by R-phase precipitation and NiTi are additive.

C. Toughness:

The literature on these materials does not typically discuss fracture mode or attempt to discuss fracture mechanisms; as a result normally all that is available are compositions, toughness results, tensile properties, reported heat treatments and some limited microstructural data. Based on those considerations and our own work in the area, primarily on the effects of copper particle strengthening and strengthening by NiAl precipitation we have come to the following conclusions. First, all precipitation hardened systems exhibit a minimum in toughness at aging temperatures coincident with the onset of precipitation strengthening. This effect is most pronounced when large copper additions are used. We attribute this behavior to particle shearing which promotes local strain softening and strain localization. Second, utilizing one type of precipitation hardening particle such as NiAl has limits in terms of the amount of hardening which can be achieved without reaching unacceptable levels of toughness. For example we observe that when the composition is otherwise held constant and the aluminum, for example, is increased, the strength increases with the square root of the aluminum content and the Charpy impact energy decreases at a somewhat faster rate. One could achieve yield strengths in alloys such as PH13-8 as high as 250ksi by using higher aluminum levels but the toughness becomes quite low. Similar comments could be made about strengthening by NiTi. We favor using two or more particle hardening systems so that the particles in one of the systems are no longer shearable and these particles encourage wavy rather than planar slip. This is possibly one reason that the alloys AFC260, which utilizes secondary hardening and R-phase strengthening, and Ultrafort 401, which utilizes NiTi and R-phase strengthening, have the highest levels of fracture toughness at high strength levels (see Figures 2 and 3).

Asayama[9] has carried out the most complete studies of strengthening by R-phase and how molybdenum and nickel levels influence age-hardening and toughness. The compositions studied by Asayama contained 0.05C / 12 Cr / 12Co / 0.2Ti / xNi / yMo. Asayama investigated the effects of molybdenum content at a nickel level of about 4.5 wt.% and the effects of nickel at a molybdenum content of 5 wt.% on age-hardening response and the fracture stress of side grooved flat tensile specimens. The specimen used for determining the fracture stress can, in principle, be used to determine fracture toughness but the specimens were only 3mm thick so that at the higher fracture stresses the fracture toughnesses calculated would not be valid. The compositions investigated by Asayama[9] are listed in Table II. Asayama heat treated his materials by austenitizing at 1050°C, quenching, refrigerating at -76°C for 15hours and then aging at the indicated temperatures for 4hours. The effects of nickel at a fixed molybdenum content of 5 wt.% and of molybdenum at a fixed nickel content of 4.5 wt.% on hardness and the fracture stress are shown in Figures 4 and 5, respectively. Nickel additions had the effect of removing the embrittlement trough and resulting in higher fracture stresses at a peak hardness but did not seem to significantly influence the age-hardening response. This effect of nickel one might attribute to an inherent effect of nickel (similar effects have been observed on adding nickel, palladium and platinum to alpha iron) on the ductile-to-brittle transition temperature. However, Asayama's retained austenite measurements[9], Figure 6, show that increasing the nickel increases the retained austenite content and the improvements in fracture stress associated with increasing nickel might be due to the increasing amounts of retained austenite. The results for the effect of molybdenum are seen in Figure 5 and, as expected, the age-hardening response increases with molybdenum content but additions of molybdenum also eliminate the embrittlement trough. Note that on aging to close to peak hardness the higher molybdenum levels still have the higher fracture stress even though they are somewhat harder. While Asayama does not report retained austenite levels for these alloys, it is suggested, based on the known retained austenite contents for the higher molybdenum level, that decreasing the molybdenum decreases the martensite start temperature and decreases the amount of retained austenite. Our conclusion from Asayama's work is that retained austenite can significantly influence fracture in this system.

Our results indicate also that retained austenite may be important to achieving reasonable toughness in precipitation hardened martensitic stainless steels. Consider the four alloys whose compositions are given in Table III. The four alloys, WC88, WC89, WC90 and WC91, are the same except that WC88 and WC89 contain 4 wt.% and 5 wt.% nickel respectively and both WC88 and WC89 contain 15 wt.% chromium while the alloys WC90 and WC91 contain 4 and 5 wt.% nickel respectively and both contain 12 wt.% chromium. Thus WC88 and WC90 are identical except that WC88 contains 15 wt.% chromium and WC90 contains 12 wt.% chromium. WC89 and WC91 are identical except that WC89 contains 15 wt.% chromium and WC90 contains 12 wt.% chromium. All four alloys contain 1 wt.% titanium and 2.8 wt.% molybdenum so that NiTi and R-phase strengthening are expected. The four alloys were heat treated by austenitizing at 1050°C, oil quenching, refrigerating in liquid nitrogen for 24hours and then aging for 3.16 hours. The room temperature hardnesses and Charpy impact energies are given for the four alloys as a function of aging temperature in Table III. Our primary interest was hardening response, hence we did not expect high toughness given the large austenite grain size (130µm) after austenitizing at 1050°C; however the Charpy impact values were surprising. The Charpy impact values were quite low for the alloys WC90 and WC91 for all aging temperatures. The alloy WC89 was quite soft as it contained large amounts of retained austenite. However, the alloy WC88 had a Charpy impact value of 44ft-lbs at a hardness of Rockwell-C 47.5. The retained austenite contents for these alloys have not been measured yet, but it is clear that the retained austenite content of WC88 is greater than the retained austenite contents of alloys WC90 and WC91 which had low toughness. The fracture mode for the alloys WC90 and WC91 was quasi-cleavage while the fracture modes for the alloy WC88 was ductile. Thus our results suggest that retained austenite can help lower the ductile-to-brittle transition temperature.

D. Corrosion Resistance:

A primary goal is that the alloys have excellent general corrosion resistance. Work done in the late 1960's and early 1970's indicates that when precipitation strengthened alloys containing cobalt of the types described in the background are aged the corrosion resistance is less than the corrosion resistance of the alloy in the solution-treated or as-quenched condition. This has been attributed to the formation of precipitates such as the R phase which contain chromium and a resulting decrease in the amount of chromium in the matrix. This possible explanation has never been tested. However, the extent to which corrosion resistance is reduced on aging is not clear. For some compositions in which R-phase strengthening is expected the effect is dramatic but in others excellent corrosion properties are still obtained[4].

E. Stress Corrosion Cracking Resistance:

The data from Truman's review article[11] on the stress corrosion cracking of martensitic and ferritic stainless steels indicates that the precipitation strengthened martensitic stainless steels can have resistances to stress corrosion cracking which are superior to the stress corrosion cracking resistances of low alloy steels and of high alloy secondary steels such as AF1410. K_{ISSC} values for a 3.5% NaCl solution for PH13-8 and Custom 455 are 81ksi√in and 79ksi√in, respectively. The yield strength and fracture toughness reported for PH13-8 were 208ksi and 81ksi√in and the yield strength and fracture toughness reported for Custom 455 were 246ksi and 79ksi√in. Webster reports a K_{ISSC} for AFC260 of about 45ksi√in after tempering at 538°C, but this material was not refrigerated after austenitizing so that the yield strength was fairly low, only about 200ksi, even though the ultimate tensile strength was 260ksi. This result does suggest that the use of R-phase strengthening does not preclude good resistance to stress corrosion cracking resistance. Given that R phase strengthening is often associated with a degradation of corrosion resistance and of stress corrosion cracking resistance one can never be sure that a new martensitic stainless steel will have stress corrosion cracking resistance in salt water comparable to PH13-8 and Custom 455. However, the excellent stress corrosion cracking resistance of martensitic stainless steels such as PH13-8 and Custom 455 compared to low alloy steels such as 300M and even high alloy steels such as AF1410 suggests that high chromium contents (chromium actually in solid solution) may favor high stress corrosion cracking resistance.

VI. Issues:

In formulating an approach to developing a new ultra-high strength steel a considerable body of information is needed. Certainly, given the work in this area, there is enough information to begin to make guesses about useful compositions. However, the information is, in some areas, very incomplete, primarily because this class of complex materials has never received the intensive effort that other ultra high strength steel systems have received. As an example of this consider the experience of Boeing with PH15-5; Boeing recently found large variations in the toughness of this material and were stunned to find that the effects of microstructure on the toughness of PH15-5 is a subject which has never been really been addressed even though the alloy has been in existence for many years and remains to be widely used.

A. Phase Stability:

One would prefer compositions which permit the alloy to be austenitic to fairly low temperatures so that a fine grain austenite free from delta ferrite can be established prior to quenching to form martensite and which do not result in large volume fractions of retained austenite as the retained austenite can lower strength, particularly yield strength. These concerns, the requirement that the alloy should contain as much chromium as possible (hopefully at least 15 wt.%), and the necessity of achieving, by some mechanism, the required strength place severe restriction on the choice of alloying additions. Essentially the problem is this: given a desired chromium level how do we

balance austenite and ferrite stabilizers to permit low austenitizing temperatures without introducing delta ferrite and achieve the desired strength without depressing the martensite start temperature too much. There are phase diagrams, studies to assess the potency of various elements as ferrite and austenite stabilizers and data on the effects of alloying additions on martensite start (M_S) temperatures. However, these data are often incomplete or incorrect. Hull has examined the effects of a number of alloying elements in stabilizing austenite and ferrite using the method of nickel and chromium equivalents. He assigns to cobalt a nickel equivalent of 0.41. That is one weight per-cent cobalt will have the same effect as 0.41 wt.% nickel in stabilizing austenite. Other workers suggest that cobalt is a stronger austenite stabilizer than suggested by Hull. Part of the problem is that the effect of cobalt on stabilizing austenite does not appear to be linear so that if one examined the effect of cobalt as an austenite stabilizer over small ranges of cobalt variations at low and high cobalt levels one might come to very different conclusions as to the nickel equivalent for cobalt. Further, one source states that aluminum will reduce the martensite start temperature by 306°C per wt.% of aluminum; I doubt if this is the case. In addition, the effect of cobalt on the martensite start temperature is quite unusual. In some systems cobalt raises the martensite start temperature and in other systems it reduces it and in other systems it has a very small effect. Hammond's results for the effects of alloying additions on the M_S temperature, Figure 7, appear to be the most reliable for low carbon stainless steels but he reports no data for aluminum.

B. Selection and Evaluation of Strengthening Systems:

If one had to choose an alloy system immediately and hope to slightly modify this system to achieve the design goals, the obvious choice would be the alloy AFC260. This alloy has acceptable levels of toughness and stress corrosion cracking resistance and one would need to simply increase the strength a small amount. This may not be possible. The composition of AFC260 is given in Table I. This alloy is strengthened, presumably, by secondary hardening and by particles associated with R-phase strengthening. It is very difficult to imagine how the strength of this alloy could be increased by alloying modification without pushing the martensite start temperature too low. For example, one could increase the molybdenum and/or cobalt to increase the amount of R-phase strengthening, but additions of both elements would lower the martensite start temperature and the effect of cobalt on the martensite start temperature is particularly strong when the alloy contains carbon. Suppose one wanted to increase the strength by introducing NiAl precipitation; in this case the nickel would have to be increased from 2 to at least 4 wt.% and aluminum added and this modification would probably require reductions in the molybdenum and/or cobalt content and hence in a reduction in the strengthening by the R-phase process. As noted earlier the only method that I can think of to increase the strength of AFC260 is to age at 500°C to 525°C for aging times longer than the 4 hours presently used; based on the data of Asayama[10] this might increase the hardness by up to 2 Rockwell-C points.

I presently feel that the most productive approach would be to ignore the use of secondary hardening and explore the use of two or more particle strengthening processes in low-carbon alloys. At the present time I would rule out using copper additions unless the paper by Lambert et al. on the effects of copper to low carbon alloys containing 14 wt.% chromium and 20 wt.% cobalt contains provocative new results. Therefore I am presently considering only precipitation strengthening by NiAl, NiTi and the so-called R-phase.

Strengthening by NiAl and NiTi depends on the amount of nickel in the alloy at fixed titanium or aluminum levels. However, there are no quantitative studies of this effect. This information is important as, according to Hammond, in low carbon stainless steels nickel decreases the M_S temperature by 39°C per wt.% while cobalt reduces the M_S by 5°C per wt.%. This result coupled with studies of the capacities of nickel and cobalt to eliminate delta-ferrite indicates that cobalt is to be preferred to nickel as an austenite stabilizer when one considers the effects of nickel and cobalt on the M_S temperature. Therefore, I would prefer to use cobalt as the primary austenite stabilizer and use only the amount of nickel required to achieve the required strength (this approach has to be

balanced with the beneficial effect of nickel in solid solution on the ductile-to-brittle transition temperature). Therefore, It would be useful to determine the effect of nickel content on the strengthening by NiAl and NiTi.

Second, a study of R-phase strengthening would be desirable. I am unaware of any attempt to systematically investigate the effects of both cobalt and molybdenum on the intensity of the R-phase strengthening for reasonable aging times. The only study of this type is the work by Asayama[9] referred to earlier who investigated the effects of molybdenum on R-phase strengthening at a fixed cobalt level: this work was flawed by the incorporation of NiTi strengthening in all of the alloys and a rather high and variable carbon content; one of the alloys had a carbon level of almost 0.10wt.% and the carbon levels were typically 0.05wt.%. The interest here is not only in the degree of strengthening but in the precipitates actually responsible for the hardening. Typically the structures of such systems have been studied after aging at high temperatures. It would be useful to know the chromium contents of the precipitates after aging at reasonable temperatures and times as one could then estimate the chromium remaining in solid solution and available for corrosion protection.

Also, it would be useful to investigate the degree to which the NiTi and NiAl strengthening are additive to the strengthening due to the so-called R-phase strengthening.

C. Toughness and Ductile-to-Brittle Transition Temperature:

While having exceptionally high upper-shelf toughness is not a paramount concern as long as the toughness is adequate (say at least 75ksi√in) it is critical that the ductile-to-brittle transition temperature be reasonably low. One might ask if data currently available indicates which strengthening mechanism might be more favorable from the standpoint of achieving these goals. That is would strengthening by NiTi be more favorable to fracture resistance than strengthening by R-phase. Further, for a given strengthening mechanism, which mix of elements required for strengthening would be the best. For example, for R-phase strengthening should one use high cobalt and low molybdenum or would low cobalt and high molybdenum produce better toughness.

Attempting to resolve this issue from the literature is not possible. For example, there is little fractography available so we do not know if the toughness results refer to upper shelf toughness or to mixed-mode behavior; if the toughness is very low it seems reasonable to assume to quasi-cleavage fracture. To consider a specific case compare the alloys X-15 and X-23. Some properties for these two alloys are seen in Figure 2. X-15 is reported to have a yield strength of 215ksi, an ultimate tensile strength of 235ksi and a room temperature fracture toughness of about 35ksi√in. X-23 is reported to have a yield strength of about 237ksi, an ultimate tensile strength of 258ksi and a fracture toughness of 70ksi√in. Does this mean that using higher molybdenum and lower cobalt is to be preferred to using low molybdenum and high cobalt from the standpoint of toughness? Well, there is the complication that X-23 contains 7 wt.% nickel which should help to lower the ductile-to-brittle transition temperature. Further, Hammond's data suggests that the M_S temperature of X-23 is 20°C and the M_S temperature of X-15 is 196°C; these M_S temperatures suggest that X-23 may contain a substantial amount of retained austenite and X-15 would contain almost no retained austenite. We have measured the retained austenite content for an alloy very similar to X-15 and that alloy contains less than 0.25 volume % retained austenite in the as-quenched condition. As another example consider the alloy Custom 455 which has a yield strength of 235ksi and an ultimate tensile strength of 245ksi and a room temperature Charpy impact energy of 9 ft-lbs after aging at about 482°C. Could the low impact energy of this material be partly due to a low retained austenite content given that Hammond's data suggests the M_S temperature of the alloy is 167°C?

Given the fact that austenite content can have a significant effect on the toughness of these materials, most likely through its effect on the ductile-to-brittle transition temperature, one might

ask how much austenite is required. Will any austenite do or is retained austenite more beneficial than austenite formed during reversion? For a given type of austenite is the volume fraction of the austenite the critical factor or is the chemical composition of the austenite important by influencing mechanical stability of the austenite?

A concern with introducing austenite in the structure to lower the ductile-to-brittle transition temperature is the effect of retained austenite on strength. Retained austenite would be expected to lower strength, although the effect of retained austenite on yield strength appears to much stronger than its effect on the ultimate tensile strength. An alloy with an ultimate tensile strength of 275ksi and a yield strength of 190ksi would not be desirable. Therefore, any study of the effects of retained austenite on the fracture resistance of these materials should not depend on hardness measurements but should include the effect of austenite on tensile properties including yield and ultimate tensile strengths.

It would be useful to examine the effects of different strengthening mechanism on upper shelf toughness and ductile-to-brittle transition temperature for fixed nickel and austenite contents and constant strength. Such experiments may help to resolve the issue of which strengthening approach would most favor useful levels of toughness.

D. Corrosion Resistance and Stress Corrosion Cracking Resistance:

The first goal is to determine if the corrosion resistance is sufficient for the anticipated application. The second is to determine the extent to which the cobalt or the precipitation of particles associated with R-phase strengthening reduce corrosion resistance and whether this decrease in corrosion resistance is due to simply a decrease in the matrix chromium content. This would involve examining corrosion resistance as a function of aging temperature and of time. One should assess corrosion resistance and stress corrosion cracking resistance of new alloys once it was felt that alloys having the required strength and toughness were feasible.

VII. Proposed Research:

The long term objective of our work in this area is the development of an ultra-high strength stainless steel for use in landing gear which has properties described in Section II. Our initial objective is determine compositions which have the desired strength and fracture resistance and which we believe may have the desired corrosion resistance. Thus our approach is use as high a chromium level as possible and, consistent with these high levels of chromium, attempt to achieve the strength and fracture resistance required.

That achieving the desired strength levels is possible is beyond doubt. The aging curves of Asayama[10] in Figure 8 indicate that with R-phase strengthening one can achieve hardnesses of almost Rockwell-C 55 (Rockwell-C 53 without sacrificing notch tensile strength) using a composition of 0.024C/4.28Ni/12Cr/12Co/5.1Mo. In addition, the data of Diderrich et al.[8] seen in Figure 9 indicate that by using 20Co and 5Mo one can achieve hardnesses of Rockwell-C 59.5 which is a Vickers hardness of 680. The data of Bungardt et al[15]. suggests that in a low carbon 12Cr/8Ni/5Co/2Mo/1.4Ti alloy one can achieve yield strengths of about 270ksi and ultimate tensile strengths of over 280ksi.

The issue then is how to achieve the desired strength and maintain the desired fracture resistance. One could potentially use strengthening by NiAl, NiTi or by R-phase either singly or in combination. Our conclusion based on the alloys WC88, WC89, WC90 and WC91 is that to achieve the desired strength at a chromium level of 15 wt.% using combined strengthening mechanisms will be difficult. The reason for this is that strengthening by NiTi is such a strong function of nickel level; increasing the nickel level above 4 wt.% to increase the strengthening by NiTi and still adding enough molybdenum and cobalt to obtain substantial R-phase strengthening

would result in amounts of retained austenite which would be unacceptable. As the austenite content is increased the strength, particularly the yield strength, will be decreased below the desired levels. Unfortunately, the amount of austenite which would be excessive is not known although I suspect the maximum amount of austenite which can be introduced cannot be much more than 10 volume per-cent.

Therefore, it is planned to investigate strengthening by NiTi and R-phase separately. Ideally these experiments would investigate for each strengthening mechanism the effects of retained austenite on fracture resistance and tensile properties when the composition of the austenite is held fixed and all systems contained the same amounts of nickel in solid solution. We have focused on this approach because the amount of retained austenite seems to have a strong effect on the ductile-to-brittle transition temperature but the amount of retained austenite cannot become so large that the yield strength becomes too low even if the ultimate tensile strength remains high.

While I do not believe that we can control the compositions to achieve the ideal experiments the following systems are proposed for study.

A. Strengthening by NiTi:

The intention here is to examine the effects of austenite on the fracture resistance and tensile properties of a system strengthened by NiTi. The initial composition would be 15Cr, 8.5Ni and 1.4 Ti. Now this system, using Hulls results for the chromium equivalent for titanium, would have a chromium equivalent of 18.08. According to the results of Hull there should be no ferrite in the structure if the nickel equivalent is 13 or more. The nickel equivalent for cobalt given by Hull of 0.41 (which our experience suggests is conservative) would imply a cobalt requirement of 11 wt.%. We will use 12 wt. % to bring the cobalt levels a little closer to those in some of the R-phase systems. Thus the first composition in this system would be 15Cr/8.5Ni/12Co/1.4Ti. The data of Hammond suggests that this alloy will have an M_S temperature of 5.5°C. The M_S temperature of our alloy WC88 was predicted by the Hammond data to be 6°C. It is intended then to vary the M_S temperature in this system by varying the chromium content. The initial alloys proposed and their predicted M_S temperatures are listed below:

Composition	M_S temperature (°C)
12Cr/12Co/8.5Ni/1.4Ti	92.5
13Cr/12Co/8.5Ni/1.4Ti	63.5
14Cr/12Co/8.5Ni/1.4Ti	34.5
14.5Cr/12Co/8.5Ni/1.4Ti	20
15Cr/12Co/8.5Ni/1.4Ti	5.5

It may be that the compositions will need to be modified slightly depending on the results obtained from say the first two heats melted which would be the 15Cr and 12Cr versions. However, the intention is to vary the retained austenite content by varying the chromium level. For this series the goal is to determine how the amount of retained austenite influences the ductile-to-brittle transition temperature and the tensile properties as a function of aging temperature and time. In addition, the effect of austenitizing temperature on austenite grain size will be investigated and, to the extent

possible, the effects of grain size on ductile-to-brittle transition temperature investigated. The M_S temperatures will be determined. If this alloy system seems promising we can return to the system and attempt to possibly reduce the cobalt (to reduce cost) and increase the chromium depending on the results.

B. Strengthening by R-Phase:

Suppose the system contains 15 wt.% chromium and 5 wt.% molybdenum then the system would contain, based on Hull's results, a total chromium equivalent of 21.05. To be free of delta ferrite the system should contain a nickel equivalent of 15 wt.%. If no nickel is added then, based on Hull's nickel equivalent for cobalt of 0.41, this would require a cobalt level of 36 wt.%. If the nickel equivalent is 0.8 the required cobalt would be 18.75 wt.%. So there may be some problems here in terms of delta ferrite even if modest amounts of nickel are added, both to put nickel in solid solution and to vary the M_S temperatures. However, Asayama's 12Cr/12Co/4.28Ni/5.1Mo alloy had a chromium equivalent of 18.17 wt.%; Hull's data suggests that this alloy would require a nickel equivalent of 13 or more to avoid delta ferrite. Using Hull's nickel equivalent for cobalt of 0.41 Asayama's alloy has a nickel equivalent of 9.2 wt.%. If we assume that the nickel equivalent of 1 wt.% cobalt is 0.8wt.% then Asayama's alloy has a nickel equivalent of 14 wt.%. Thus at this point I am reasonably comfortable with using 0.8 as the nickel equivalent for cobalt.

I propose three systems involving R-phase strengthening. The first is essentially that explored by Asayama[9]. The other two have 15 wt.% chromium and 15Co/5Mo and 20Co/4Mo for strengthening. All three systems should achieve hardnesses suggested by an ultimate tensile strength of 275ksi. In each system the M_S temperatures will be varied by varying the nickel contents. The alloys proposed for each system and the M_S temperature for each system predicted by Hammond's results are given below:

B.1 12Cr/12Co/5Mo:

Composition	M_S temperature ($^{\circ}\text{C}$)
12Cr/12Co/5Mo/3Ni	127
12Cr/12Co/5Mo/4Ni	88
12Cr/12Co/5Mo/4.5Ni	68.5
12Cr/12Co/5Mo/5Ni	49
12Cr/12Co/5Mo/5.5Ni	32.5
12Cr/12Co/5Mo/6Ni	13

B.2 15Cr/15Co/5Mo:

Composition	M_S temperature ($^{\circ}\text{C}$)
15Cr/15Co/5Mo/2Ni	100

15Cr/15Co/5Mo/3Ni	61
15Cr/15Co/5Mo/3.5Ni	41.5
15Cr/15Co/5Mo/4Ni	22

B.3 15Cr/20Co/4Mo:

Composition	M _S temperature (°C)
15Cr/20Co/4Mo/1Ni	114
15Cr/20Co/4Mo/2Ni	75
15Cr/20Co/4Mo/2.5Ni	55.5
15Cr/20Co/4Mo/3Ni	36
15Cr/20Co/4Mo/3.5Ni	16.5

The first two alloys melted for each series would be the high and low nickel alloys and the results from these alloys may suggest changes in compositions, as Hammond's data may not be totally reliable. However, the intention is to vary the retained austenite content in each series by varying the nickel level. For each series the goal is to determine how the amount of retained austenite influences the ductile-to-brittle transition temperature and the tensile properties as a function of aging temperature and time. In addition, the effect of austenitizing temperature on austenite grain size and ferrite content will be investigated and, to the extent possible, the effects of grain size on ductile-to-brittle transition temperature investigated. The M_S temperatures will be determined.

VIII. Summary of Proposed Research:

Four precipitation hardening systems are proposed for study. The amount of retained austenite in each system will be varied by varying composition. The effect of retained austenite content on ductile-to-brittle transition temperature and tensile properties will be examined as a function of aging temperature in each system. The effect of austenitizing temperature on grain size will be investigated, God willing that the alloys can be etched, and the effects of grain size on ductile-to-brittle transition temperature investigated.

If one or more of the systems appears suitable in terms of strength and fracture resistance then a number of further issues need to be resolved. These would include a more complete study of strengthening processes if R-phase is involved, processing for grain size control, homogenization methods, control of inclusion distributions for maximum upper-shelf toughness and studies of corrosion resistance and stress corrosion cracking resistance and of fatigue behavior.

Teledyne-Allvac has supported this work and will continue to do so as long as the results appear encouraging. In order to carry out, either concurrent or future corrosion and stress corrosion cracking studies, Teledyne-Allvac will make larger heats of the proposed compositions so that corrosion work can be carried out on heats already characterized to some extent. Up to now the

heats have been 25 pound heats produced by vacuum induction melting. The heat size will be scaled up to at least 50 pounds.

IX. Results:

A. Alloys Prepared:

Four approaches to precipitation strengthening, one utilizing NiTi and three utilizing R-phase, were proposed for study. In each system the goal was to vary the amount of retained austenite and determine influence of retained austenite on the ductile-to-brittle transition temperature. The alloys prepared to date in each of the systems are those with a heat identification number next to the nominal composition in the lists below.

All of the alloys were prepared as 50 pound ingots made by vacuum induction melting followed by vacuum arc re-melting. Each alloy was rolled to flat bar. The carbon levels were kept to 0.002 wt. % carbon in order to eliminate secondary hardening as a strengthening mechanism; this precaution was taken because it was felt that the strengths of the alloys investigated by Asayama, which contained 0.025 to 0.05 wt. % carbon, were substantially influenced by secondary hardening.

1. Strengthening by NiTi:

Alloy	Composition	M_s temperature (°C)
	12Cr/12Co/8.5Ni/1.6Ti	92.5
WD55	13Cr/12Co/8.5Ni/1.6Ti	63.5
WD56	14Cr/12Co/8.5Ni/1.6Ti	34.5
WD57	14.5Cr/12Co/8.5Ni/1.6Ti	20
WD58	15Cr/12Co/8.5Ni/1.6Ti	5.5

2. Strengthening by R-Phase:

2.b 12Cr/12Co/5Mo:

Alloy	Composition	M _S temperature (°C)
	12Cr/12Co/5Mo/3Ni	127
	12Cr/12Co/5Mo/4Ni	88
WD59	12Cr/12Co/5Mo/4.5Ni	68.5
WD60	12Cr/12Co/5Mo/5Ni	49
WD61	12Cr/12Co/5Mo/5.5Ni	32.5
WD62	12Cr/12Co/5Mo/6Ni	13
WE09	12Cr/12Co/5Mo/6.5Ni	-6.5
WE10	12Cr/12Co/5Mo/7Ni	-25

2.c 15Cr/15Co/5Mo:

Alloy	Composition	M _S temperature (°C)
	15Cr/15Co/5Mo/2Ni	100
WE11	15Cr/15Co/5Mo/2.5Ni	81.5
WE12	15Cr/15Co/5Mo/3Ni	61
	15Cr/15Co/5Mo/3.5Ni	41.5
WD63	15Cr/15Co/5Mo/4Ni	22

2.d 15Cr/20Co/4Mo:

Alloy	Composition	M _S temperature (°C)
	15Cr/20Co/4Mo/1Ni	114
WE13	15Cr/20Co/4Mo/1.5Ni	95.5
WE14	15Cr/20Co/4Mo/2Ni	75
	15Cr/20Co/4Mo/2.5Ni	55.5
	15Cr/20Co/4Mo/3Ni	36
WD64	15Cr/20Co/4Mo/3.5Ni	16.5

B. Mechanical Properties and Austenite Contents:

All of the alloys have been heat treated by austenitizing at 1050°C for one hour and then refrigerating in liquid nitrogen for 5 hours and then tempering at the indicated temperatures and times, if the samples were tempered.

All tensile specimens were of the longitudinal orientation and the Charpy impact specimens were of the longitudinal-transverse orientation.

The austenite contents of the alloys have been determined in the quenched and refrigerated condition for all of the alloys and as a function of tempering temperature (for tempering times of 3.16 hours) for the alloys WD59, WD60, WD61 and WD62. The austenite contents in the as-quenched and refrigerated condition are given along with the hardness and Charpy impact data in Tables IV through Table VII. The austenite contents of WD59, WD60, WD61 and WD62 are given in Table XIII as a function of tempering temperature.

B.1 Mechanical Properties After Aging for 3.16 hours:

The hardnesses and room temperature Charpy impact energies after tempering for 3.16 hours are given in Table IV, Table V, Table VI and Table VII. The tensile properties of alloys WD59, WD60, WD61 and WD62 after tempering for 3.16 hours are given as a function of tempering temperature in Table VIII.

B.1.a. Strengthening by NiTi:

The four alloys prepared were WD55, WD56, WD57 and WD58 which contained 13, 14, 14.5 and 15 wt. % chromium, respectively. The volume % of retained austenite in the alloys WD55, WD56, WD57 and WD58 after oil quenching and refrigerating were 14.7, 39.9, 62.4 and 77.6, respectively. Thus all except alloy WD55 contained too much retained austenite. The alloy WD55 contained 14.7 wt. % retained austenite. The properties of these alloys are summarized in Table IV. The alloy WD55 alloy achieved a peak hardness on aging of only R_c 46.8. Furthermore, the room temperature Charpy impact energies for this material were extremely low unless the alloy is tempered at 550°C and at this tempering temperature the hardness is only R_c 41.7. As the alloy WD55 had such poor toughness at room temperature, even though it contained 14.7 wt. % retained austenite, and because the strength of the alloy is so low, it is not believed that useful room temperature toughness or a sufficiently low ductile-to-brittle transition temperature can be achieved with this alloy at the desired strength levels. Unless our later results suggest that retained austenite is actually harmful to the ductile-to-brittle transition temperature our intention is to stop work on this alloy system.

B.1.b. Strengthening by R-Phase:

B.1.b.1 12Cr/12Co/5Mo:

For this system the four alloys initially melted, WD59, WD60, WD61 and WD62, contained 4.5, 5, 5.5 and 6 wt. % nickel, respectively. The four alloys WD59, WD60, WD61 and WD62, contained 0.05, 0.19, 0.79 and 6.2 volume % retained austenite respectively. Two alloys, WE09 and WE10, containing 6.5 and 7 wt. % nickel, respectively, and contained 18.6 and 36.8 volume % retained austenite, respectively. For aging times of 3.16 hours the hardness and room temperature Charpy impact energy are given as a function of tempering temperature in Table V. The room temperature tensile properties of the four alloys are given as a function of tempering temperature (for 3.16 hour tempering times) in Table VIII.

The first point is that the maximum hardness achieved for the alloy WD59 is about 6 R_C less than the hardness reported by Asayama for a similar alloy and for similar aging times and temperatures. A heat was produced of the same composition as WD59 except that the new heat contained 0.025 wt. % carbon, a carbon level similar to that employed by Asayama. The hardnesses achieved by this new alloy were more similar to those achieved by the Asayama alloy of the same composition. The results suggest that the hardnesses of these materials can be substantially influenced by small carbon additions.

Second, increasing the nickel content improved the room temperature Charpy impact energy, most dramatically at aging temperatures below the temperature at which the peak hardness is achieved. The improvements in toughness associated with increasing the nickel content were greatest as the nickel was increased from 5.5 to 6 wt. %. Increasing the nickel from 5.5 to 6 wt. % increased the retained austenite from about 0.79 volume % to about 5.1 volume %.

Third, increasing the nickel content in this system modestly increased the peak hardness which can be achieved by aging and decreases the tempering temperature at which the peak precipitation hardness is observed. However, once the nickel content is increased above 6 wt. % the hardness decreases with increasing nickel content. This decrease in hardness is presumably due to increased amounts of austenite, both retained and reverted, in the structure. The hardnesses after tempering at 500°C of the 6 wt. % nickel alloy and of the alloy containing 6.5 wt. % nickel were R_C 47.6 and 46, respectively. Presently, this decrease in hardness is attributed to an increased amount of retained austenite. The hardnesses after tempering at 525°C of the 6 wt. % nickel and 6.5 wt. % nickel alloys were R_C 48.7 and 45.1, respectively. On tempering at 525°C the decrease in hardness associated with increasing the nickel content from 6 to 6.5 wt. % is much greater than that observed after tempering at 500°C and this larger decrease in hardness is attributed to significant reversion of martensite to austenite in the alloy containing 6.5 wt. % nickel alloy during tempering at 525°C.

The room temperature tensile properties of the four alloys, WD59, WD60, WD61 and WD62 are given as a function of tempering temperature (for 3.16hour tempering times) in Table VIII. One unusual aspect of the tensile data is that for higher aging temperatures the reduction-in-areas are not as large as one might expect on the basis of the Charpy impact energies. Also the reduction-in-areas obtained for higher aging temperatures decrease as the nickel content is increased, while the Charpy impact energies tend to increase as the nickel content is increased

B.1.b.2 15Cr/15Co/5Mo:

Three alloys have been melted for this system, WE11, WE12 and WD63, containing 2.5, 3.0 and 4.0 wt. % nickel, respectively. The alloy WD63 was melted first and contained 32.2 volume % retained austenite after quenching and refrigerating. This was too much retained austenite and the alloys WE11 and WE12 were then melted. The retained austenite contents of WE11 and WE12 after quenching and refrigerating, were 8.3 and 21.2 volume %, respectively. The hardness achieved by the alloy WE11 after tempering at 525°C was R_C 49.3 and the room temperature Charpy impact energy of WE11 after tempering at 525°C was 33 foot-pounds. The data for this alloy series are in Table VI.

B.1.b.3 15Cr/20Co/4Mo:

Three alloys have been melted for this system, WE13, WE14 and WD64, containing 1.5, 2 and 3.5 wt. % nickel, respectively. The alloy WD64 was melted first and contained 59.6 volume % retained austenite after quenching and refrigerating. This was too much retained austenite and the alloys WE13 and WE14 were then melted. The retained austenite contents of WE13 and WE14 after quenching and refrigerating, were 16.4 and 31.0 volume %, respectively. The hardness achieved by the alloy WE13 after tempering at 525°C was R_C 49.2 and the room temperature

Charpy impact energy of WE13 after tempering at 525°C was 31.5 foot-pounds. The data for this alloy series are in Table VII.

B.2 Mechanical Properties for Longer Tempering Times:

The hardnesses, room temperature Charpy impact energies and room temperature tensile properties of the alloys WD59, WD60, WD61 and WD62 have been determined as a function of tempering time for the tempering temperatures of 500°C, 525°C and 550°C. The hardness and Charpy impact energies obtained for longer tempering times are given in Table X and the tensile data for longer tempering times are in Table IX. The primary observations are that longer aging times can substantially increase the strength and hardness but there is a concomitant decrease in Charpy impact energy and reduction-in-area. This embrittlement does not appear to be associated with just the increase in strength level as for a given strength level the Charpy impact energy and tensile ductility are higher when the strength is achieved by tempering for short times than when the strength is achieved by tempering for longer times at a lower temperature.

B.3 Effect of Test Temperature on Charpy Impact Energies:

The Charpy impact energy has been determined as a function of test temperature for the four alloys WD59, WD60, WD61 and WD62 for tempering temperatures of 500°C and 525°C for tempering times of 3.16 hours. These results are given in tabular form in Table XI and plotted in Figures 10 and 11. The results show that for both the tempered at 500°C and 525°C microstructures the alloy WD62 has much better toughness at low test temperatures than do the other three alloys. In fact, for the tempered at 525°C microstructure of WD62 the ductile-to-brittle-transition temperature (DBTT) appears to be about -75°C while the ductile-to-brittle-transition temperatures for the other three alloys appear to be -10°C or higher. The tempered at 525°C for 3.16 hours microstructure of the alloy WD62 has a yield strength of 205.5ksi and an ultimate tensile strength of 231.2ksi at room temperature. This DBTT is much lower than that of any currently available martensitic stainless steels which have comparable strength levels. For example, the DBTT of PH13-8 tempered at 525°C for 3.16 hours is about -15°C.

C. Effect of austenitizing Temperature:

As summarized in Table XII the effect of austenitizing temperature on hardness and Charpy impact energy after tempering for 3.16 hours at 525°C was investigated for austenitizing temperatures of 900°C, 950°C, 1000°C, and 1050°C for an austenitizing time of one hour for the alloys WE11 and WE13. The results indicate that lower austenitizing temperatures are not to be used as lower austenitizing temperatures can result in much lower hardness or toughness.

D. Effect of Tempering Temperature on Austenite Content:

The austenite in the as-quenched and refrigerated condition has been measured for all of the alloys prepared. The austenite content has been determined as a function of tempering temperature for the alloys WD59, WD60, WD61 and WD62. The austenite content is given as a function of tempering temperature in Table XIII.

X. Discussion:

The purpose of this work was to assess the effects of the amount of retained austenite content on the ductile-to-brittle transition temperature of martensitic precipitation strengthened stainless steels for four different precipitation strengthening systems, one utilizing NiTi strengthening and three

utilizing R-phase strengthening. The retained austenite contents in the four systems were varied by varying composition. The austenite content in the NiTi strengthened system was varied by varying the chromium content and the austenite content in the R-phase strengthened systems was varied by varying the nickel content.

The room temperature toughness levels of the NiTi strengthened system were quite low and it was decided not to pursue this system further.

The three R-phase strengthened systems had sufficient room temperature toughness and strength to be of further interest. Of these three systems the primary focus was on the 12Cr/12Co/5Mo system. In this system there were six alloys, four of which, WD59, WD60, WD61 and WD62, were the primary focus of the work. The alloys WD59, WD60, WD61 and WD62 could, on tempering at 525°C for 3.16 hours, achieve yield strengths on the order of 210ksi and ultimate tensile strengths of 235ksi. Of greatest interest in this work was the effect of test temperature on the Charpy impact energies of the four alloys.

The effect of test temperature on the Charpy impact energy was investigated for the tempered at 500°C for 3.16 hours and the tempered at 525°C for 3.16 hours microstructures of the four alloys, WD59, WD60, WD61 and WD62. It was found that for both tempering conditions the alloy WD62 had lower ductile-to-brittle transition temperatures than the other three alloys. In fact, the ductile-to-brittle transition temperature of the tempered at 525°C for 3.16 hours microstructure of WD62 was quite low, about -75°C.

It is not clear why the ductile-to-brittle transition temperature of the tempered at 525°C for 3.16 hours microstructure of WD62 is so low. The ductile-to-brittle transition temperature is determined by the composition and the fine-scale microstructure. WD62 has half a percent nickel more than the alloy WD61; this higher nickel content could itself contribute to a lowering of the ductile-to-brittle transition temperature. The higher nickel content of WD62 could also result in fine-scale microstructural changes which help lower the ductile-to-brittle transition temperature. It is clear that WD62 has more retained austenite in the as-quenched condition than the other three alloys. It may be that this increased amount of retained austenite is sufficient to explain the low ductile-to-brittle transition temperature of WD62 tempered at 525°C for 3.16 hours. However, the retained austenite measurements indicate that there was reversion of martensite to austenite on tempering at 525°C for 3.16 hours. It may be that this reverted austenite also contributes to the low ductile-to-brittle transition temperature of this microstructure. It may also be that the half a percent nickel is sufficient to alter the fine-scale microstructure in a way not related to austenite content and that such changes also help lower the ductile-to-brittle transition temperature. At this point the authors believe the austenite content of WD62 is the most significant contributor to the low ductile-to-brittle transition temperature of the WD62 tempered at 525°C for 3.16 hours microstructure.

To examine the role of austenite content and the relative importance of retained and reverted austenite in controlling the ductile-to-brittle transition temperature the following experiments are contemplated. They would be to increase the retained austenite content by making chromium additions to the alloy WD59, which contains only 4.5 wt.% nickel. By making a series of such alloys varying in chromium content, one could vary the retained austenite content. Further, since the nickel is much reduced, one would not expect reverted austenite to form on tempering for a few hours at temperatures of 525°C or lower. If the ductile-to-brittle transition temperature was found to decrease with increasing retained austenite content for such a series of alloys then one could argue that retained austenite content exerts a significant effect on the ductile-to-brittle transition temperature. Further, if the ductile-to-brittle transition temperature was found to be about -75°C when the retained austenite content was 6 to 7 volume percent one could argue strongly that WD62 tempered at 525°C for 3.16 hours has a lower ductile-to-brittle transition temperature than WD59,

WD60 and WD61 tempered at 525°C for 3.16hours primarily because of its higher retained austenite content.

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Table I
Compositions of Some Martensitic High Strength Stainless Steels

Material	C	Cr	Ni	Co	Mo	Cu	Ti	Al	Nb	V
PH15-5	.05	15	5	-	-	3	-	-	0.3	-
PH13-8	.04	13	8	-	2.2	-	-	1.0	-	-
Custom 450	0.05	15	6.5	-	0.8	1.5	-	-	0.7	-
Custom 455	0.03	11.5	8.5	-	-	2.2	1.2	-	0.25	-
IN734	0.02	11.8	8.5	-	-	-	0.33	1.2	-	-
AFC77	0.15	14.5	-	13.5	5.0	-	-	-	-	0.5
AFC260	0.08	15.5	2	13.5	4.3	-	-	-	0.14	-
Pyromet X-15	0.01	15	-	20	2.9	-	-	-	-	-
Pyromet X-23	0.03	10	7	10	5.5	-	-	-	-	-
Ultrafort 401	0.02	12.5	7.6	5.3	2.0	-	0.8	-	-	-
Ultrafort 403	0.02	11	7.7	9	4.5	-	0.4	0.15	-	-

Table II
Compositions Investigated by Asayama [9]

Charge No.	Composition %						Analyzed charge No
	C	Cr	Ni	Co	Mo	Ti	
9, 19	0.05 0.036	12.0 11.97	3.0 2.98	12.0 12.08	5.3 5.35	0.2 0.18	19
0, 20	0.05 0.049	12.0 11.93	4.2 4.25	12.0 11.91	5.0 5.08	0.2 0.16	20
1, 21 1	0.05 0.062	12.0 11.75	4.5 4.49	12.0 12.00	5.0 5.00	0.2 0.16	31
2, 22 3, 24, 51	0.05 0.042	12.0 11.84	5.0 5.04	12.0 12.10	5.0 4.98	0.2 0.17	23
3	0.05 0.03	12.0 11.81	6.0 6.02	12.0 12.00	4.0 4.08	0.2 0.15	13
4	0.05 0.045	12.0 11.71	4.6 4.68	12.0 11.95	2.0 2.16	0.2 0.15	14
5	0.05 0.051	12.0 11.80	4.6 4.53	12.0 12.02	2.5 2.54	0.2 0.19	15
6	0.05 0.039	12.0 11.89	4.6 4.56	12.0 12.06	3.3 3.28	0.2 0.14	16
7	0.05 0.042	12.0 11.89	4.6 4.48	12.0 11.93	4.0 3.96	0.2 0.16	17
8	0.05 0.09	12.0 11.72	4.6 4.44	12.0 12.26	4.4 4.41	0.2 0.16	18

Upper column : Nominal composition, Lower column : Analyzed composition

Table III

Chromium Effects: R-Phase and NiTi Combined

Material	C	Ni	Co	Cr	Mn	Mo	V	Si	Ti	Al	Cu	S	P
WC88	.003	4.0	14.9	14.8	.01	2.74	.01	.01	.90	.01	.01	26	<30
WC89	.004	5.0	14.9	14.8	.01	2.74	.01	.01	.91	.01	.01	28	<30
WC90	.006	4.0	15.0	11.8	.01	2.77	.01	.01	.93	.01	.01	30	<30
WC91	.004	5.0	15.0	11.7	.01	2.75	.01	.01	.92	.01	.01	26	<30

Material	A.Q.	450°C	475°C	500°C	525°C	550°C
WC88						
Hardness(R_C)	32.0	45.4	45.1	45.5	47.1	47.5
Charpy(ft-lbs)		34	46	41	28	44
WC89						
Hardness(R_C)	16.5	25.1	25.9	26.9	26.0	27.0
Charpy(ft-lbs)		>120	111	115	112	120
WC90						
Hardness(R_C)	25.6	38.9	41.0	43.8	44.9	45.6
Charpy(ft-lbs)		2	2	1.5	1	2
WC91						
Hardness(R_C)	26.0	41.8	44.0	47.0	48.0	47.6
Charpy(ft-lbs)		2	1	1	2	2

Table IV**Strengthening by NiTi****Hardness, Charpy Impact Energy and As-Quenched Retained Austenite Content**

Alloy	A.Q.	450	475	500	525	550
WD55 ret. aust.	14.7					
Charpy(ft-lbs)		2.5	2.5	4	5	30
Hardness(R _C)		45.8	46.0	46.8	46.2	41.7
WD56 ret. aust.	39.9					
Charpy(ft-lbs)		65	55	57	73	90
Hardness(R _C)		24.0	23.6	24.7	24.1	21.1
WD57 ret. aust.	62.4					
Charpy(ft-lbs)		117	109	98	168	177
Hardness(R _C)						
WD58 ret. aust.	77.6					
Charpy(ft-lbs)		197	167	158	199	198
Hardness(R _C)						

Table V

Strengthening by R-Phase: 12Cr/12Co/5Mo

Hardness, Charpy Impact Energy and As-Quenched Retained Austenite Content

Alloy	A.Q.	450	475	500	525	550
WD59 ret. aust.	0.5					
Charpy(ft-lbs)		22.5	10.5	12	26	15
Hardness(R _C)		39.5	42.0	43.4	45.6	47.7
WD60 ret. aust.	1.6					
Charpy(ft-lbs)		10.5	14	20	24.5	17
Hardness(R _C)		41.1	43.1	45.1	47.0	47.6
WD61 ret. aust.	1.8					
Charpy(ft-lbs)		9.5	18	22	23.5	21.5
Hardness(R _C)		42.5	44.8	46.4	48.6	48.0
WD62 ret. aust.	5.1					
Charpy(ft-lbs)		36	28	37	30	25.5
Hardness(R _C)		44.1	45.9	47.6	48.7	46.8
WE09 ret. aust.	18.6					
Charpy(ft-lbs)		-	-	33	33.5	-
Hardness(R _C)		-	-	46	45.1	-
WE10 ret. aust.	36.8					
Charpy(ft-lbs)		-	-	28.5	34.5	-
Hardness(R _C)		-	-	40	38.7	-

Table VI**Strengthening by R-Phase: 15Cr/15Co/5Mo****Hardness, Charpy Impact Energy and As-Quenched Retained Austenite Content**

Alloy	A.Q.	450	475	500	525	550
WE11						
ret. aust.	8.3					
Charpy(ft-lbs)		-	-	34.5	33	28.5
Hardness(R _C)		-	-	48.9	49.3	49.8
WE12						
ret. aust.	21.2					
Charpy(ft-lbs)		-	-	36	37.5	-
Hardness(R _C)		-	-	46.5	45.9	-
WD63						
ret. aust.	32.2					
Charpy(ft-lbs)		66	53	46	47	57
Hardness(R _C)		34.4	36.6	36.9	38.5	38

Table VII**Strengthening by R-Phase: 15Cr/20Co/4Mo****Hardness, Charpy Impact Energy and As-Quenched Retained Austenite Content**

Alloy	A.Q.	450	475	500	525	550
WE13						
ret. aust.	16.4					
Charpy(ft-lbs)		-	-	26.5	31.5	33.5
Hardness(R _C)		-	-	48.8	49.2	49.4
WE14						
ret. aust.	31.0					
Charpy(ft-lbs)		-	-	39.5	40	-
Hardness(R _C)		-	-	41.5	44.1	-
WD64						
ret. aust.	59.6					
Charpy(ft-lbs)		100	99	89	87	91
Hardness(R _C)		26.8	27	28.6	28.1	26.4

Table VIII**Tensile Properties of Alloys WD59 Through WD62**

Microstructure	Yield Strength (ksi)	U.T.S (ksi)	Elongation (%)	R.A. (%)
WD59				
300-3.16hr	114.1	134.9	19	75.8
400-3.16hr	137.1	157.7	18	69.8
450-3.16hr	166.6	180.3	18	61.8
475-3.16hr	178.9	202.3	16	56.5
500-3.16hr	194.7	209.4	15	53.8
525-3.16hr	205.4	220.0	15	57.5
550-3.16hr	206.7	239.5	16	54.7
WD60				
300-3.16hr	115.7	139.7	18	74.1
400-3.16hr	142.7	161.3	18	68.5
450-3.16hr	172.4	188.1	17	57.5
475-3.16hr	187.4	199.9	17	51.1
500-3.16hr	204.9	216.0	12	48.6
525-3.16hr	214.2	228.8	14	52.5
550-3.16hr	211.3	246.2	13	45.5
WD61				
300-3.16hr	117.6	144.0	18	74.1
400-3.16hr	149.2	167.4	17	67.5
450-3.16hr	179.0	194.8	17	52.5
475-3.16hr	195.2	203.3	16	47.3
500-3.16hr	212.0	223.7	12	48.6
525-3.16hr	220.3	238.2	13	50.7
550-3.16hr	211.7	247.7	14	41.6
WD62				
300-3.16hr	119.7	150.5	20	73.6
400-3.16hr	146.6	174.0	20	66.4
450-3.16hr	188.3	207.8	18	50.8
475-3.16hr	194.2	210.3	18	48.3
500-3.16hr	209.2	225.1	15	48.0
525-3.16hr	205.5	231.2	16	49.0
550-3.16hr	190.7	233.0	16	47.4

Table IX**Strengthening by R-Phase: 12Cr/12Co/5Mo****Tensile Properties of Alloys WD59 Through WD62**

Microstructure	Yield Strength (ksi)	U.T.S (ksi)	Elongation (%)	R.A. (%)
WD59				
500-3.16hr	194.7	209.4	15	53.8
500-10hr	213.7	226.3	13	45.2
500-31.6hr	232.8	250.8	14	39.6
500-100hr	232.8	264.4	11	28.9
525-3.16hr	205.4	220.0	15	57.5
525-10hr	225.1	249.1	13	43.4
550-3.16hr	206.7	239.5	16	54.7
WD60				
500-3.16hr	204.9	216.0	12	48.6
500-10hr	223.3	235.0	11	40.9
500-31.6hr	235.8	256.1	11	33.0
500-100hr	231.9	262.8	10	24.1
525-3.16hr	214.2	228.8	14	52.5
525-10hr	229.4	257.1	12	33.3
550-3.16hr	211.3	246.2	13	45.5
WD61				
500-3.16hr	212.0	223.7	12	48.6
500-10hr	231.4	244.8	11	41.0
500-31.6hr	242.1	261.2	11	32.8
500-100hr	232.2	262.9	11	22.7
525-3.16hr	220.3	238.2	13	50.7
525-10hr	228.6	256.2	10	34.6
550-3.16hr	211.7	247.7	14	41.6
WD62				
500-3.16hr	209.2	225.1	15	48.0
500-10hr	224.3	244.7	14	42.4
500-31.6hr	214.4	247.3	12	34.3
500-100hr	206.4	242.8	12	25.0
525-3.16hr	205.5	231.2	16	49.0
525-10hr	209.7	246.6	12	34.5
550-3.16hr	190.7	233.0	16	47.4

Table X

**Effect of Tempering Time for Alloys WD59, WD60, WD61 and WD62
on Hardness and Charpy Impact Energy**

Alloy	Tempering Temperature (C)	Tempering Time			
		3.16hr	10hr	31.6hr	100hr
WD59	500				
	Charpy(ft-lbs)	12	9.7	4.1	6.8
	Hardness(R _C)	43.4	47.0	50.6	51.8
	525				
	Charpy(ft-lbs)	26	11	8.8	
	Hardness(R _C)	45.6	49.1	49.7	
	550				
	Charpy(ft-lbs)	15	12.1	8.5	
WD60		47.7	47.6	48.0	
	500				
	Charpy(ft-lbs)	20	-	6.2	6.7
	Hardness(R _C)	45.1	-	51.7	52.1
	525				
	Charpy(ft-lbs)	24.5	11.7	7.0	
	Hardness(R _C)	47.0	50.3	51.4	
	550				
WD61		47.6	49.4	48.4	
	500				
	Charpy(ft-lbs)	22	14.6	7.2	8.1
	Hardness(R _C)	46.4	49.9	51.2	51.9
	525				
	Charpy(ft-lbs)	23.5	10.5	8.5	
	Hardness(R _C)	48.6	50.3	50.4	
	550				
WD62		48.0	48.1	46.7	
	500				
	Charpy(ft-lbs)	37	8.7	14.0	12.2
	Hardness(R _C)	47.6	51.8	51.9	50.1
	525				
	Charpy(ft-lbs)	30	18.8	11.8	
	Hardness(R _C)	48.7	50.4	50.4	
	550				
	Charpy(ft-lbs)	25.5	19.4	13.3	
	Hardness(R _C)	46.8	48.3	46.3	

Table XI**Effect of Test Temperature on Charpy Impact Energy**

Aging Temp. Alloy	Test Temperature					
	-85°C	-40°C	-10°C	20°C	60°C	100°C
500°C Age						
WD59						
Charpy(ft-lbs)	-	3	8	12	22	37.5
WD60						
Charpy(ft-lbs)	-	6	10.5	20	23.5	35
WD61						
Charpy(ft-lbs)	-	8.5	18	22	34.5	39.5
WD62						
Charpy(ft-lbs)	9	14	35	37	-	35.5
525°C Age						
WD59						
Charpy(ft-lbs)	-	9.5	10	26	27	43.5
WD50						
Charpy(ft-lbs)	-	7	18	24.5	26	40
WD61						
Charpy(ft-lbs)	-	7.5	19	23.5	30	31
WD62						
Charpy(ft-lbs)	15.5	25	-	30	-	32

Table XII**Effect of Austenitizing Temperature on Hardness and Toughness**

Alloy	900 OQ/525	950 OQ/525	1000 OQ/525	1050 OQ/525	1050 OQLN/525
WE11					
ret. aust.					
Charpy(ft-lbs)	7	17	28.5	37	33
Hardness(R _C)	48.8	47	48.3	49	49.3
WE13					
ret. aust.					
Charpy(ft-lbs)	32	31	31.5	34	31.5
Hardness(R _C)	44.5	46.1	46.6	48.4	49.2

Table XIII**Austenite Content as a Function of Tempering Temperature**

Alloy	Tempering Temperature					
	A.Q.	300°C	400°C	500°C	525°C	550°C
WD59						
ret. aust.	6.22	7.24	8.7	8.6	10.9	20.6
WD60						
ret. aust.	0.79	0.67	0.64	0.81	1.1	3.5
WD61						
ret. aust.	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
WD62						
ret. aust.	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5

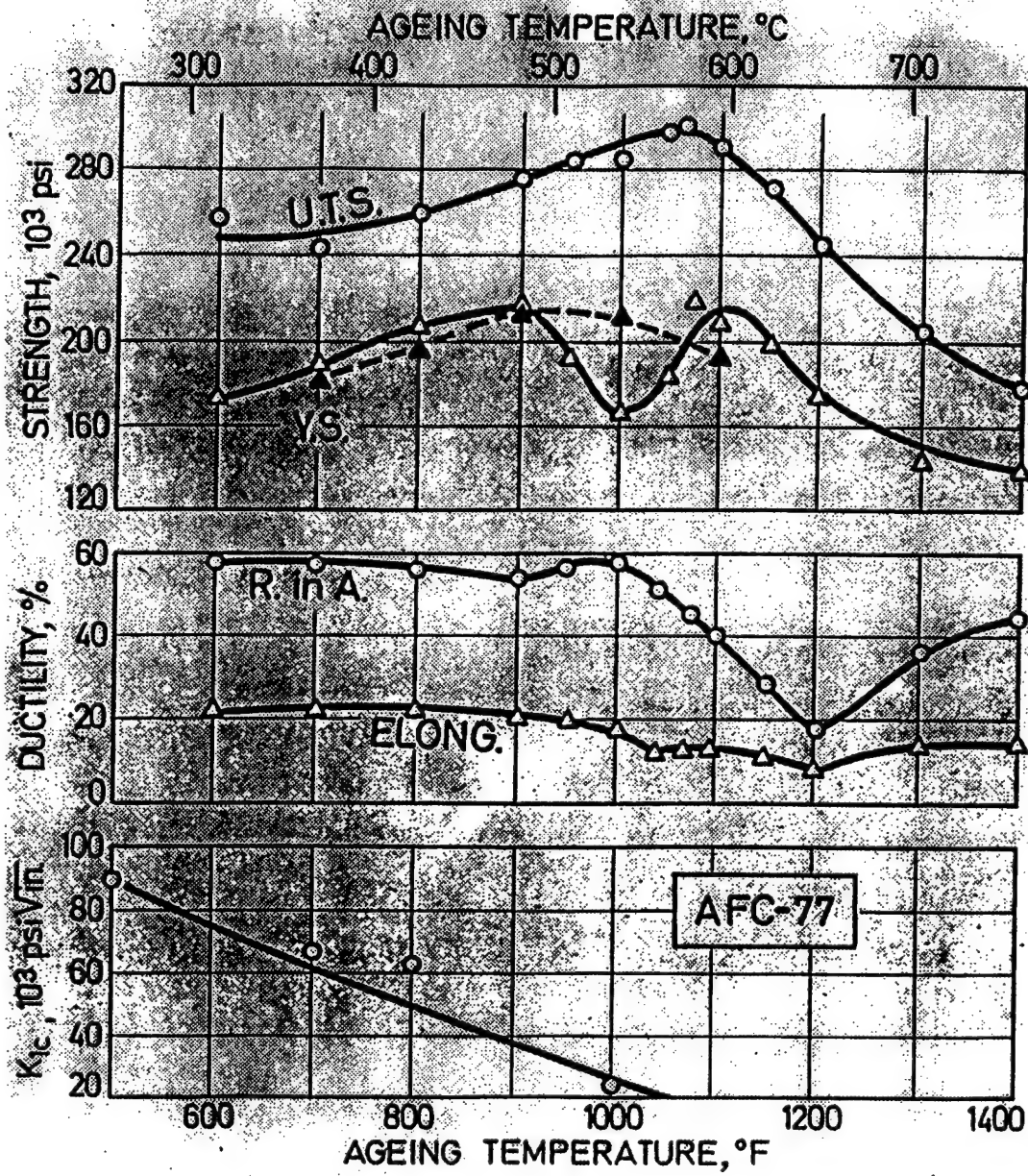


Figure 1: Mechanical properties of AFC77 plotted as a function of tempering temperature[4].

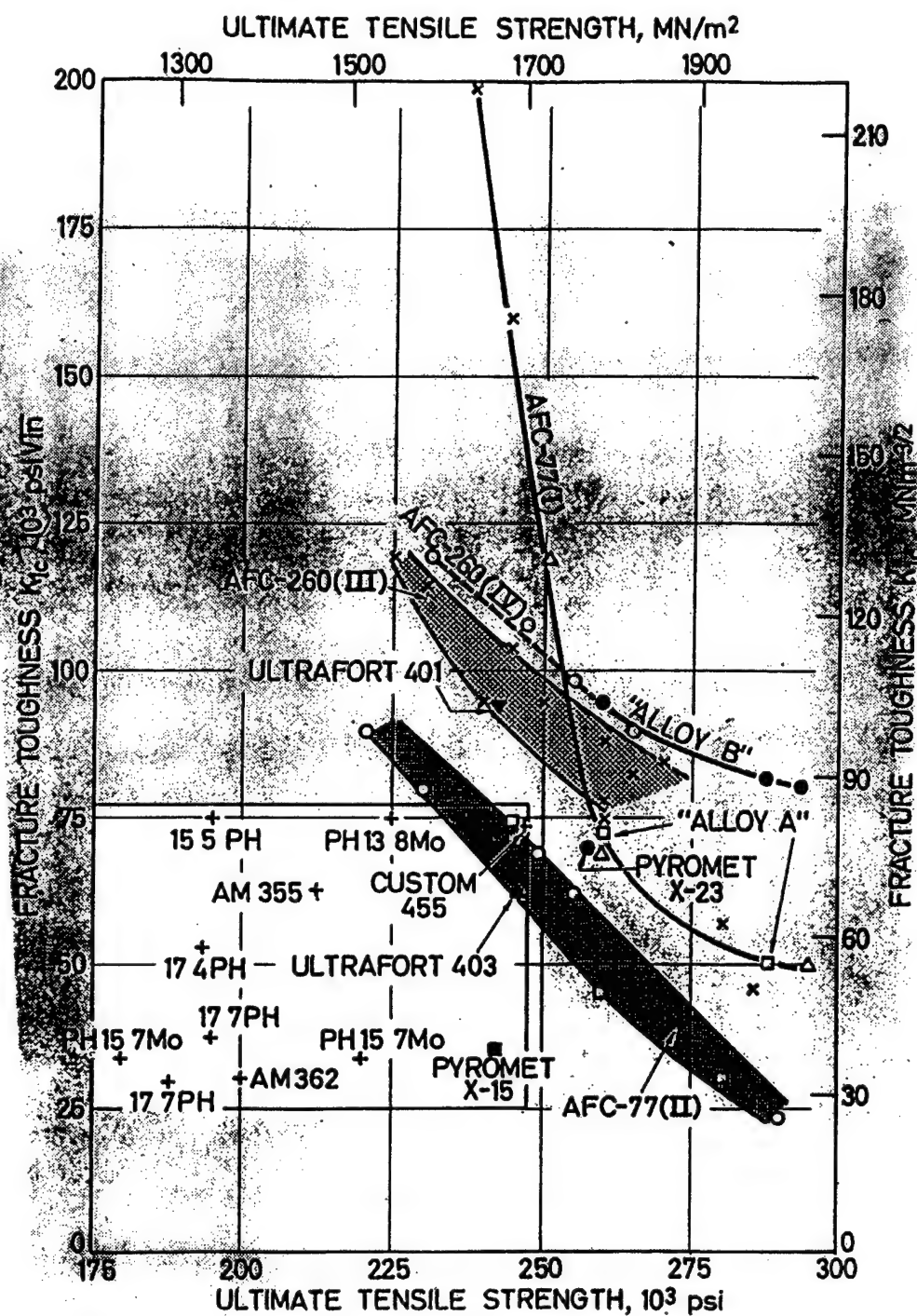


Figure 2: The fracture toughness plotted as a function of ultimate tensile strength for a number of ultra-high strength martensitic stainless steels[4].

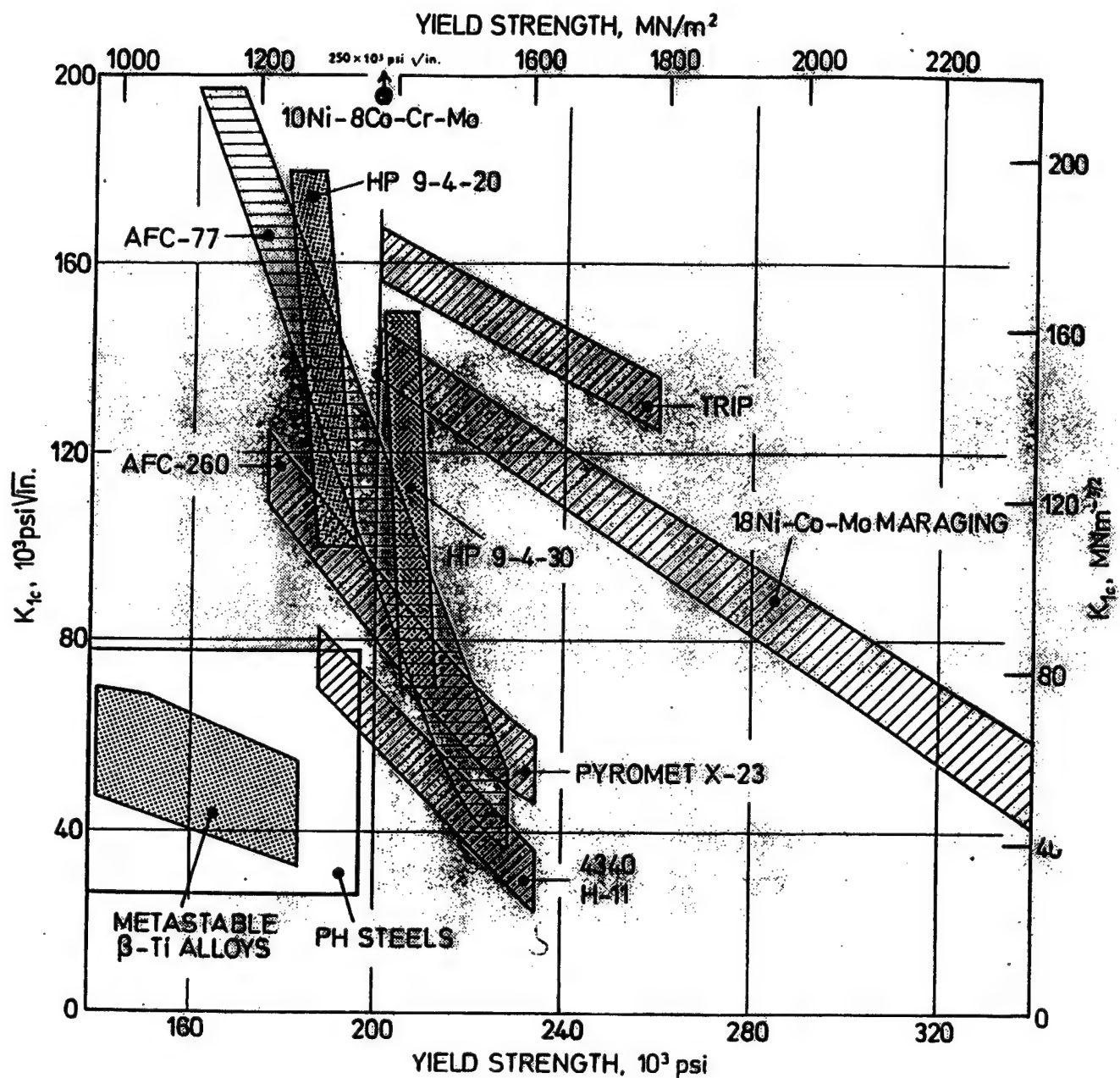


Figure 3: The fracture toughness plotted as a function of yield strength for a number of ultra-high strength martensitic steels[4].

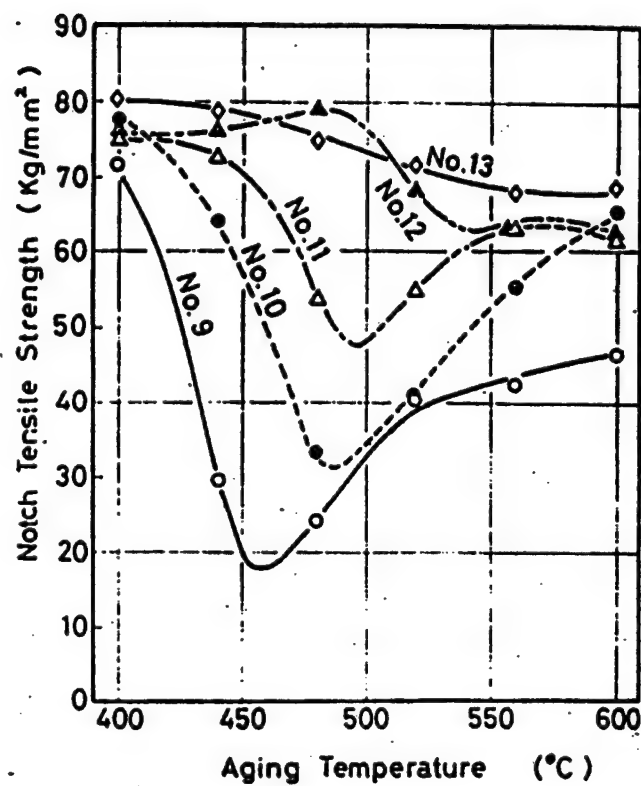
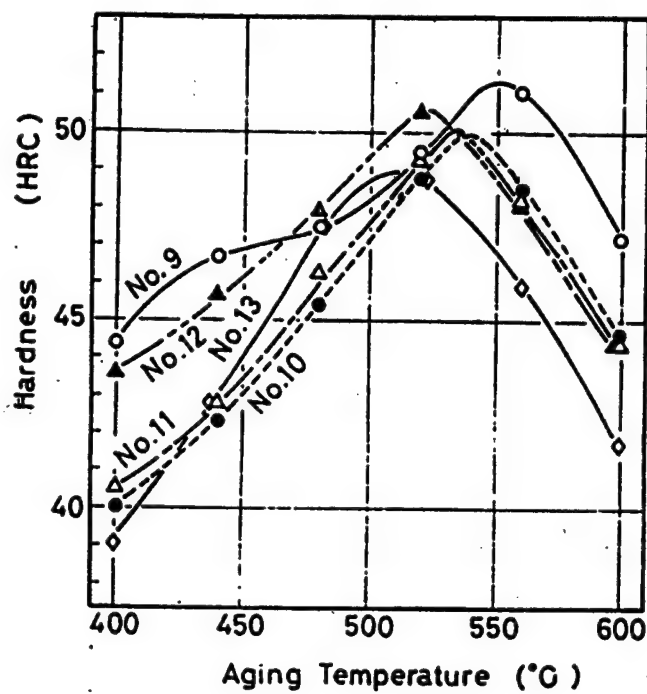


Figure 4: Asayama's results for the effect of nickel on aging response and fracture stress obtained from side cracked flat tensile specimens[9].

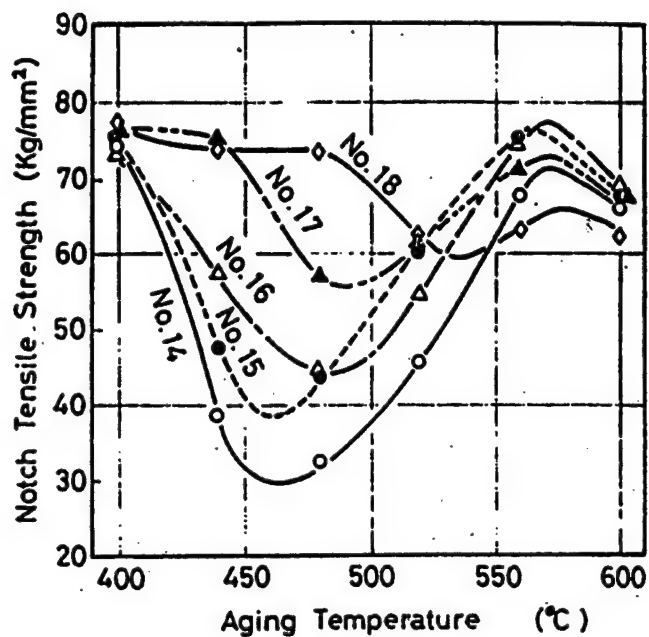
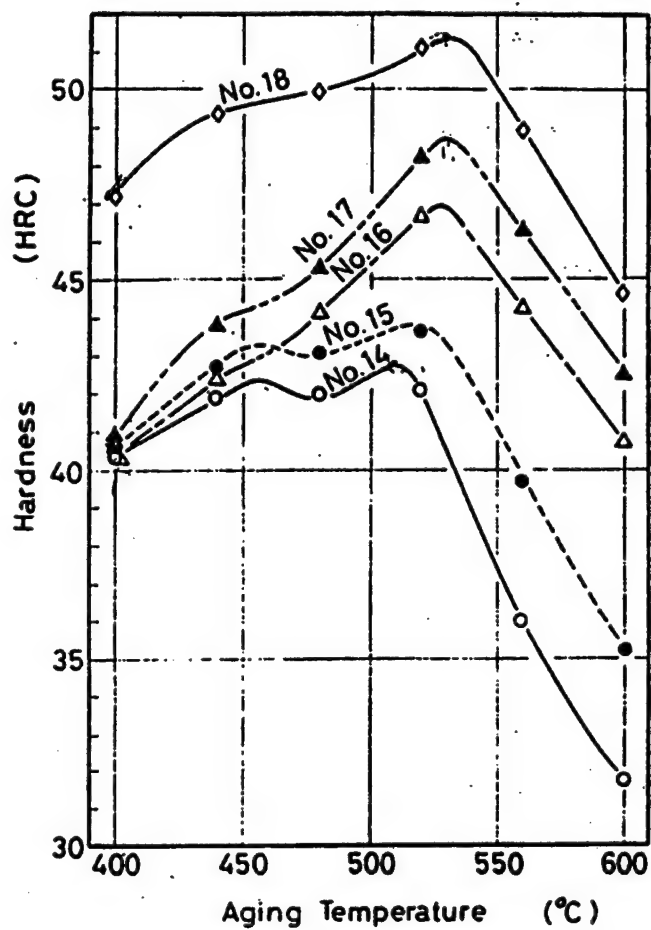


Figure 5: Asayama's results for the effect of molybdenum on aging response and fracture stress obtained from side cracked flat tensile specimens[9].

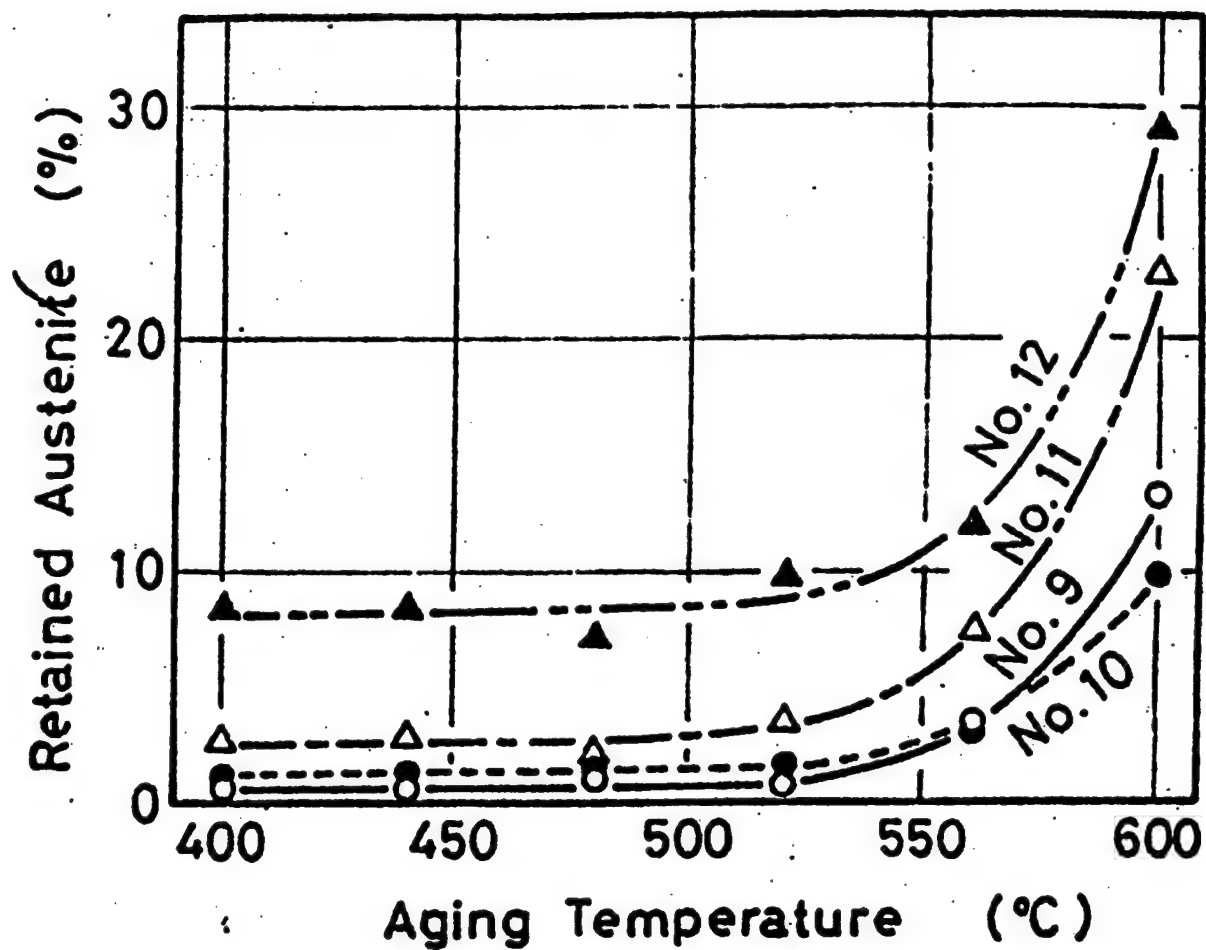


Figure 6: Asayama's results[9] for the effect of nickel content and aging temperature on retained austenite content. Heat numbers are defined in Table II. The austenite level increases with nickel content.

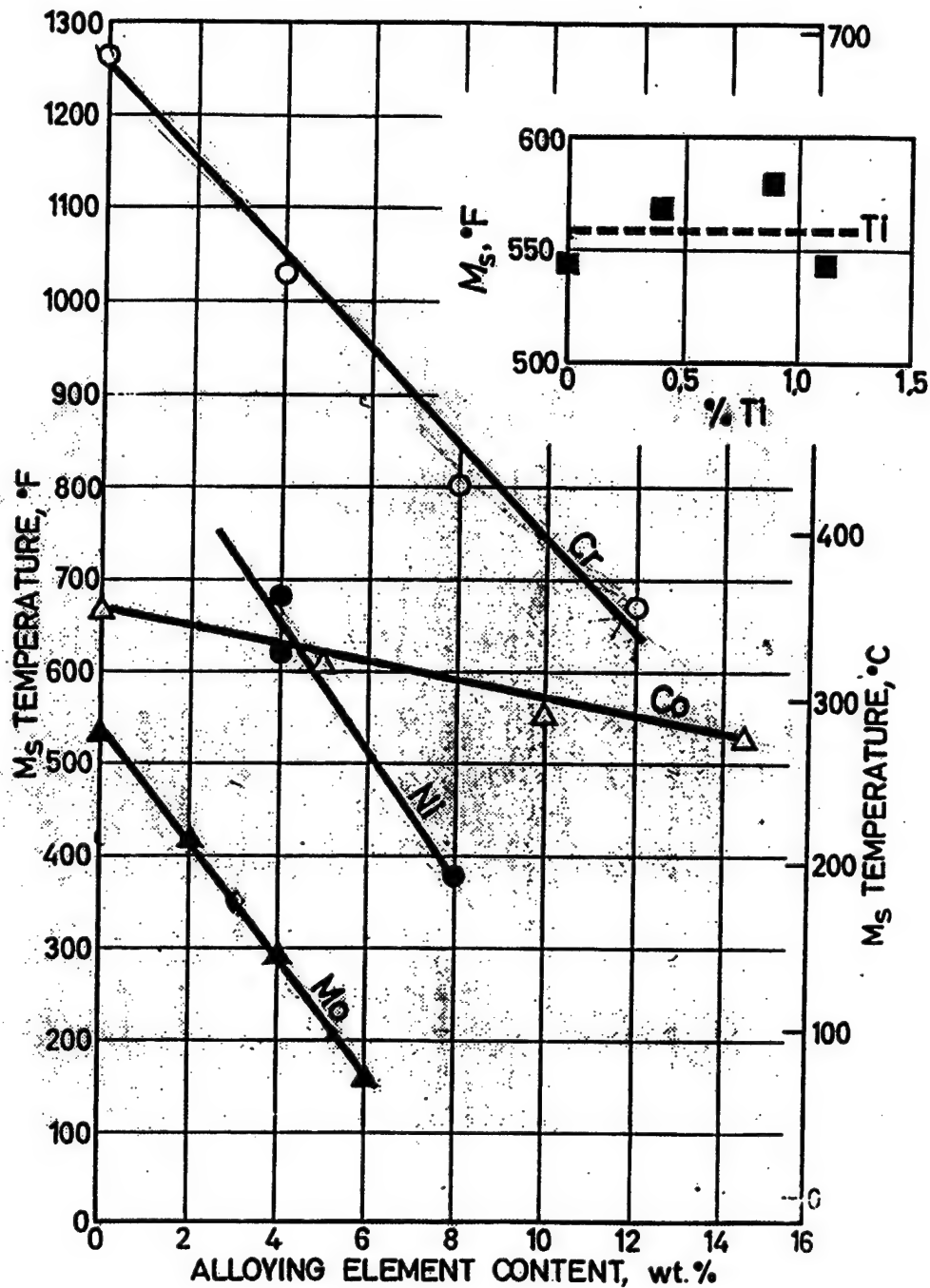


Figure 7: Hammond's results for the effects of chromium, nickel, molybdenum, cobalt and titanium on the martensite start temperature for low carbon steels. Hammond states that if the alloy contains molybdenum than titanium reduces the martensite start temperature 55°C per weight per-cent[13].

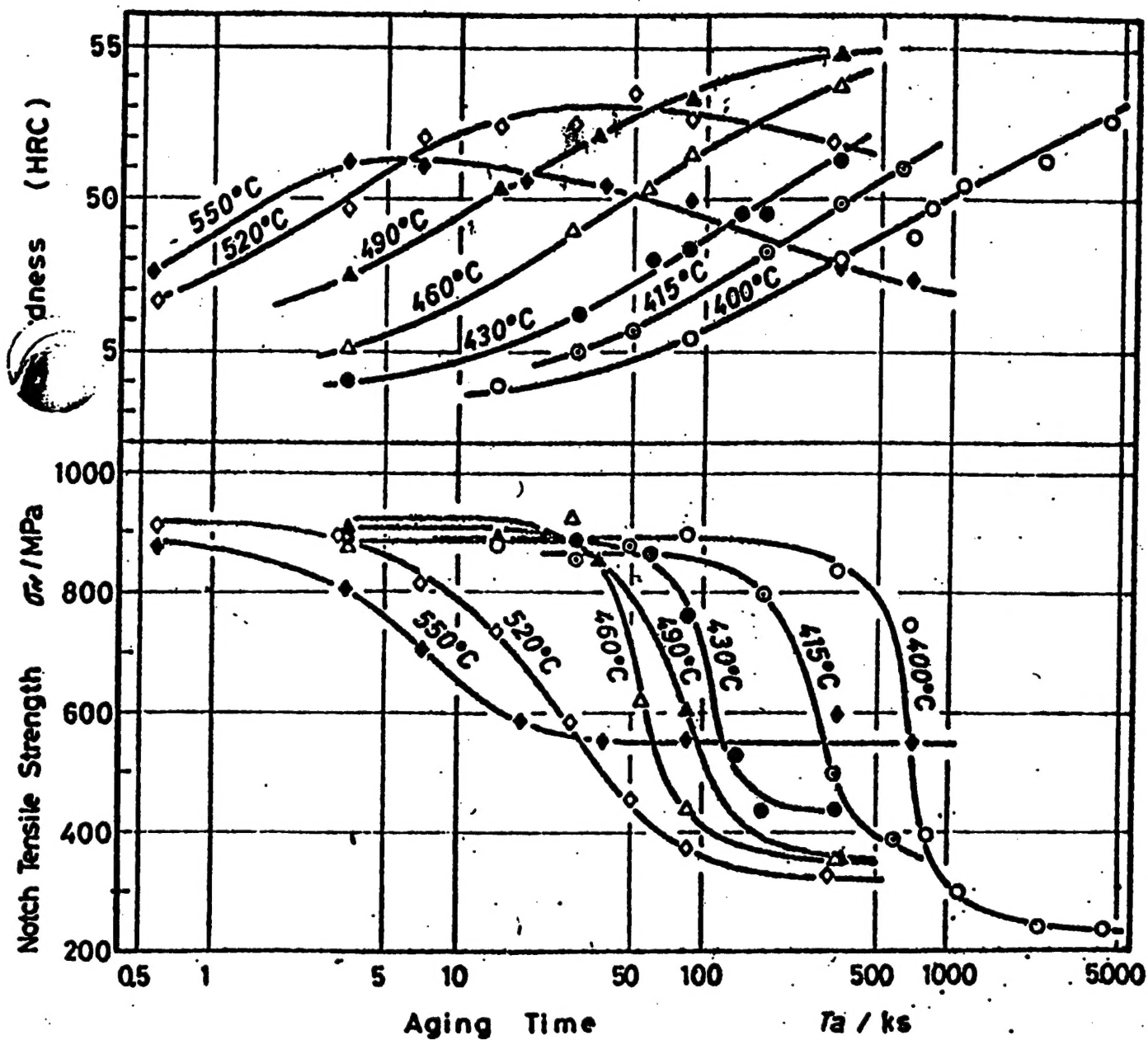


Figure 8: The effects of aging time and temperature on the hardness and fracture stress for Asayama's 12Cr/12Co/4.28Ni/5.1Mo alloy[10].

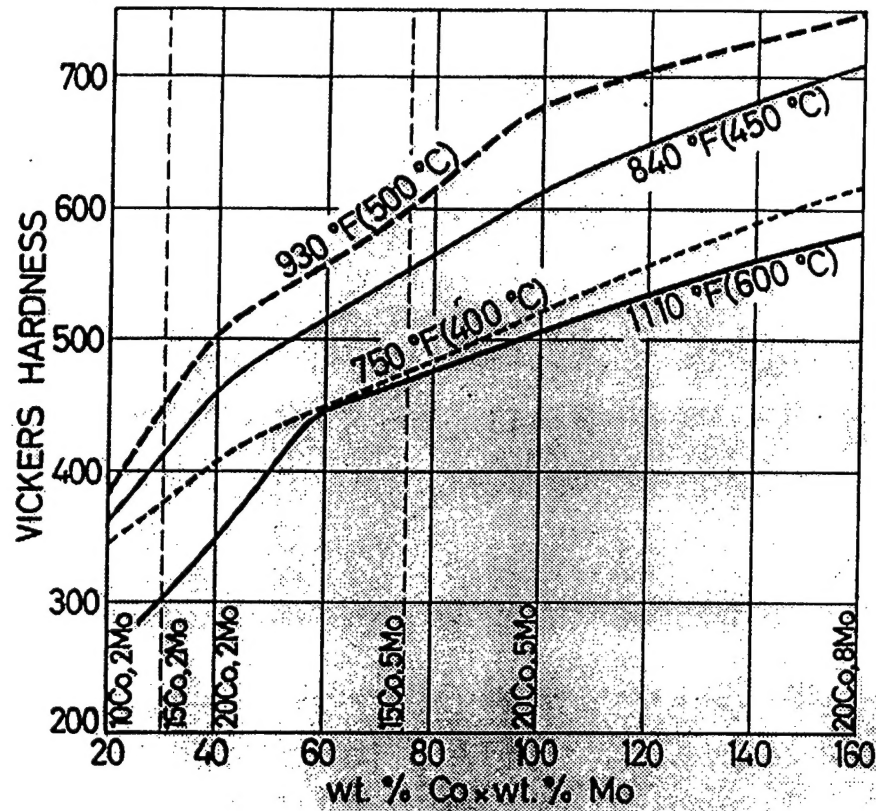


Figure 9: The results of Dederrich et al. showing the effects of molybdenum and cobalt on the hardness for indicated aging temperatures for aging times of 128 hours for a low carbon steels containing 13 wt. % chromium[7].

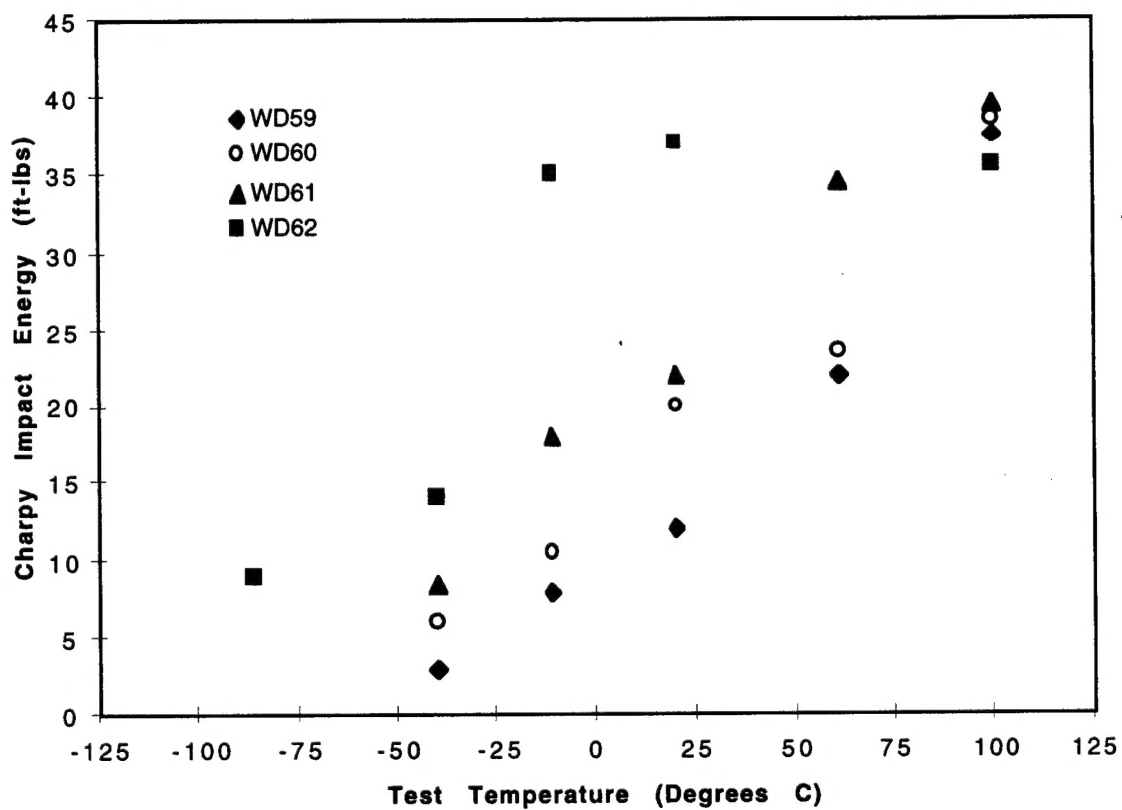


Figure 10: Charpy impact energy plotted as a function of test temperature for alloys WD59, WD60, WD61 and WD62 tempered at 500°C for 3.16 hours.

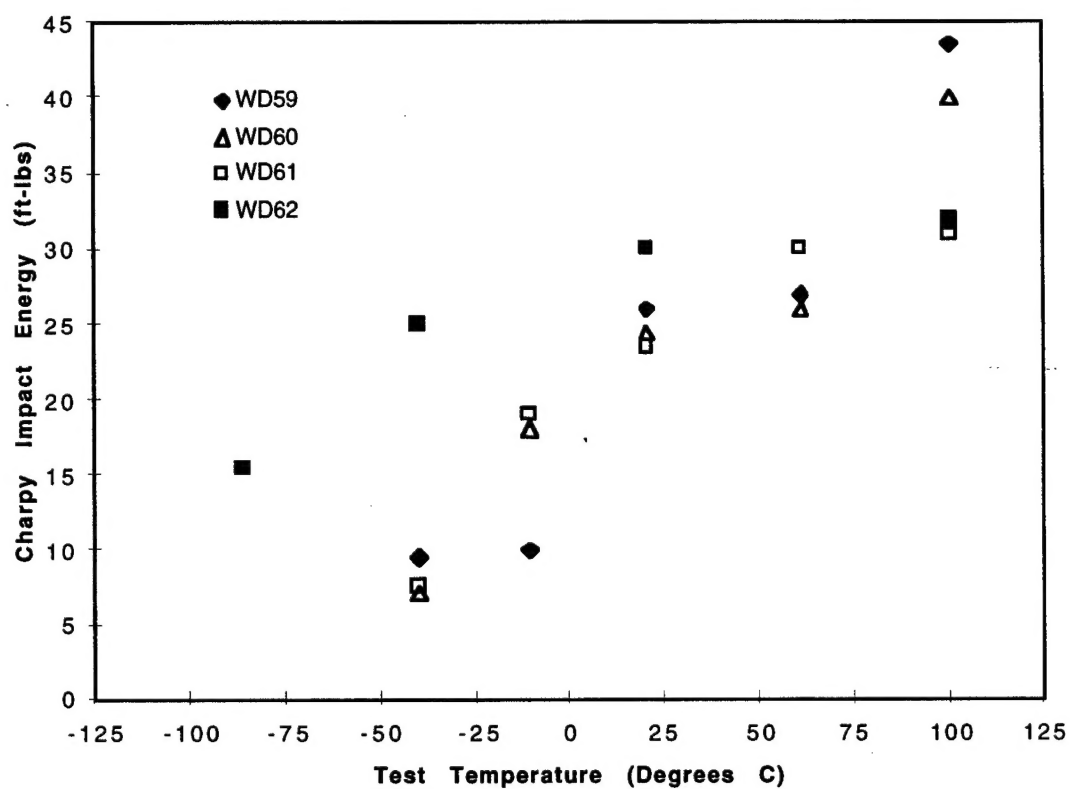


Figure 11: Charpy impact energy plotted as a function of test temperature for alloys WD59, WD60, WD61 and WD62 tempered at 525°C for 3.16hours.